AMERICAN SOCIETY
for
TESTING MATERIALS

(Reg. U. S. Pat. Office)

PROCEEDINGS

VOLUME 48
1948

COMMITTEE REPORTS
TECHNICAL PAPERS

Published by the
AMERICAN SOCIETY FOR TESTING MATERIALS
1916 Race St., Philadelphia 3, Pa.

Copyright, 1949
by the
American Society for Testing Materials

Printed in Baltimore, U. S. A
February, 1949

FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which include papers presented at the Annual Meeting or special meetings of the Society, and those accepted for publication in the ASTM BULLETIN or as Special Technical Publications.

A list of all of the Special Technical Publications published by the Society since its incorporation is given on the last two pages of this volume. The list will be supplemented annually.

Nothing contained in any publication of the American Society for Testing Materials is to be construed as granting any right, by implication or otherwise, for manufacture, sale, or use in connection with any method, apparatus, or product covered by Letters Patent, nor as insuring anyone against liability for infringement of Letters Patent.

The Society is not responsible, as a body, for the statements
and opinions advanced in this publication

CONTENTS

PROCEEDINGS

PAGE

Summary of Proceedings of the Fifty-first Annual Meeting 1

Annual Address by the President—T. A. Boyd: "If Doctor Marburg Came Back Today" 26

Annual Report of the Board of Directors 35

- Appendix I. Report of the Auditors for the Fiscal Year, January 1 to December 31, 1947 58
- Appendix II. Report of Administrative Committee on Standards 69
- Appendix III. Report of Administrative Committee on Research 73
- Appendix IV. Report of Administrative Committee on Papers and Publications 76
- Appendix V. Report of Administrative Committee on Ultimate Consumer Goods 84
- Appendix VI. Report of Administrative Committee on Simulated Service Testing 86
- Appendix VII. Report of Administrative Committee on District Activities 88

COMMITTEE REPORTS

Ferrous Metals

Report of Committee A-1 on Steel 90

- Appendix. Recommendations Affecting Standards on Steel 95

Report of Committee A-3 on Cast Iron 102

Report of Committee A-5 on Corrosion of Iron and Steel 104

- Report of Subcommittee XIV on Inspection of Black and Galvanized Sheets 108
- Report of Subcommittee XV on Field Tests of Wire and Wire Products 114
- Report of Subcommittee XVI on Field Tests and Inspection of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products 119

Report of Committee A-6 on Magnetic Properties 125

Report of Committee A-7 on Malleable-Iron Castings 130

Report of Committee A-9 on Ferro-Alloys 133

Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys 134

- Appendix. Report of Special Subcommittee on Stainless Steel Architectural Structures 137

Non-Ferrous Metals

Report of Committee B-1 on Wires for Electrical Conductors 138

Report of Committee B-2 on Non-Ferrous Metals and Alloys 143

- Appendix. A Review of the Tin Situation—Marshall W. Tuthill 146

Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys 151

- Appendix. Apparatus and Factors in Salt Fog Testing—V. M. Darsey and W. R. Cavanagh 153
- Report of Subcommittee VIII on Galvanic and Electrolytic Corrosion 167

Report of Committee B-4 on Electrical Heating, Resistance, and Related Alloys 176

Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought 178

- Appendix. Recommendations Affecting Standards for Copper and Copper Alloys 181

Report of Committee B-6 on Die-Cast Metals and Alloys 187

- Appendix. Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium 191

Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought 198

- Appendix. Tension Testing of Magnesium Alloy Tubing—A. A. Moore 205

PAGE
Report of Committee B-8 on Electrodeposited Metallic Coatings 206
Report of Committee B-9 on Metal Powders and Metal Powder Products 208
Report of Joint Committee on Filler Metal 210
Report of Joint Committee on Effect of Temperature on the Properties of Metals.. 212
  Appendix I. High Temperature Bolting Materials (Project No. 16)—Prepared by Ernest L. Robinson 214
    Discussion 236
  Appendix II. Stability of Steels as Affected by Temperature (Project No. 29)—Prepared by J. J. Kanter and E. A. Sticha 239

Cementitious, Ceramic, Concrete, and Masonry Materials

Report of Committee C-1 on Cement 244
  Appendix. Proposed Method of Test for Setting Time of Hydraulic Cement in Mortar 250
Report of Committee C-2 on Magnesium Oxychloride Cements 253
Report of Committee C-4 on Clay Pipe 254
Report of Committee C-7 on Lime 255
Report of Committee C-8 on Refractories 256
Report of Committee C-9 on Concrete and Concrete Aggregates 263
Report of Committee C-11 on Gypsum 267
Report of Committee C-12 on Mortars for Unit Masonry 269
Report of Committee C-14 on Glass and Glass Products 270
Report of Committee C-16 on Thermal Insulating Materials 271
Report of Committee C-18 on Natural Building Stone 275

Miscellaneous Materials

Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products 279
Report on the Evaluation of Surface Treatment of Steel Prior to Painting—Prepared by A. J. Eickhoff (see ASTM BULLETIN, *No. 150 January, 1948, p. 77 (TP49))*
Report of Committee D-2 on Petroleum Products and Lubricants 287
Supplementary Report of Committee D-2 on Petroleum Products and Lubricants 298
  Appendix I. Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease 303
  Appendix II. Proposed Method of Test for Separation of Residue from Butadiene 309
  Appendix III. Proposed Method of Test for Butadiene Dimer in Polymerization Grade Butadiene 312
  Appendix IV. Proposed Method of Test for Nonvolatile Residue of Polymerization Grade Butadiene 316
  Appendix V. Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases 318
  Appendix VI. Optional Use of Detonation Meter for Motor and Research Methods 328
  Appendix VII. Proposed Method of Test for Sulphur in Petroleum Oils by Quartz Tube Combustion 332
  Appendix VIII. Summary Report on Investigation of the Detonation Meter, Model 501 340
  Appendix IX. Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives 354
  Appendix X. Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products 357
  Appendix XI. Proposed Method of Analysis of Calcium and Barium Petroleum Sulfonates 367
  Appendix XII. Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry 374
Report of Committee D-3 on Gaseous Fuels 381
Report of Committee D-4 on Road and Paving Materials 383
Report of Committee D-5 on Coal and Coke 389
Report of Committee D-6 on Paper and Paper Products 395
  Appendix. Proposed Method of Wax Pick Test for Surface Strength of Paper 398
Report of Committee D-7 on Wood 400
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials. 401

PAGE
Report of Committee D-9 on Electrical Insulating Materials 402
Appendix. Recommendations Affecting Standards for Electrical Insulating Materials 410
Report of Committee D-10 on Shipping Containers 415
Report of Committee D-11 on Rubber and Rubber-Like Materials 417
Appendix. Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus 425
Report of Committee D-12 on Soaps and Other Detergents 431
Report of Committee D-13 on Textile Materials 434
Appendix. Recommendations Affecting Standards on Textile Materials 440
Report of Committee D-14 on Adhesives 445
Report of Committee D-15 on Engine Antifreezes 448
Report of Committee D-17 on Naval Stores 451
Report of Subcommittee I on Softening Point of Rosin 455
Appendix. Summary of Softening Point and Melting Point Methods Used for Testing Rosins and Resins 457
Report of Committee D-18 on Soils for Engineering Purposes 476
Report of Committee D-19 on Industrial Water 480
Appendix. Proposed Method for Identification of Types of Microorganisms in Industrial Waters 486
Proposed Method for the Sampling of Steam (see ASTM BULLETIN, *No. 151, March, 1948, p. 34)*
Report of Committee D-20 on Plastics 490
Appendix. Recommendations Affecting Standards on Plastics 494

Miscellaneous Subjects

Report of Committee E-1 on Methods of Testing 498
Report of Committee E-2 on Spectrographic Analysis 503
Report of Committee E-3 on Chemical Analysis of Metals 505
Report of Committee E-4 on Metallography 510
Report of Committee E-5 on Fire Tests of Materials and Construction 512
Report of Committee E-6 on Methods of Testing Building Constructions 515
Report of Committee E-7 on Radiographic Testing 517
Report of Committee E-8 on Nomenclature and Definitions 519
Report of Committee E-9 on Fatigue 522
Report of Committee E-11 on Quality Control of Materials 523

TECHNICAL PAPERS

Edgar Marburg Lecture

Isotopes and Their Application in the Field of Industrial Materials—P. C. Aebersold 527

Metals

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing—H. C. Cross, Ward F. Simmons, J. W. Freeman, and E. E. Reynolds 555
Discussion 589
Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson 591
Discussion 608
Atmospheric Durability of Steel Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley 610
Laboratory Corrosion Tests of Iron and Steel Pipes—George A. Ellinger, Sybil B. Marzolf and Leo J. Waldron 618
An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster 628
Discussion 645
An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—Paul G. Jones and W. J. Worley 648
Discussion 663
Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen 664
Discussion 690

PAGE

Fatigue and Static Load Tests of an Austenitic Cast Iron at Elevated Temperatures—W. Leighton Collins 696

Discussion 706

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form—H. L. Burghoff and A. I. Blank 709

Discussion 733

Tensile, Creep, and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald 737

Discussion 754

The Effect of Fatigue on Tension-Impact Resistance—William H. Hoppmann II (see ASTM BULLETIN, *No. 155, December, 1948, p. 36 (TP 241)).*

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above the Endurance Limit—James B. Duke 755

Discussion 763

An Hypothesis for the Determination of Accumulative Damage in Fatigue—F. E. Richart, Jr. and N. M. Newmark 767

Discussion 799

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis 801

Joint Discussion 825

Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee 815

Joint Discussion 825

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey 841

Discussion 853

High-Temperature Bolting Materials (Project No. 16)—Prepared by Ernest L. Robinson (Appendix I to Report of Joint Committee on Effect of Temperature on the Properties of Metals, p. 214)

Stability on Steels as Affected by Temperature (Project No. 29)—Prepared by J. J. Kanter and E. A. Sticha (Appendix II to Report of Joint Committee on Effect of Temperature on the Properties of Metals, p. 239)

A Review of the Tin Situation—Marshall W. Tuthill (Appendix to B-2 Report, p. 146)

Apparatus and Factors in Salt Fog Testing—V. M. Darsey and W. R. Cavanagh (Appendix to B-3 Report, p. 153)

Tension Testing of Magnesium Alloy Tubing—A. A. Moore (Appendix to B-7 Report, p. 205)

Quantitative Evaluation of Corrosive Conditions—W. F. Bonwitt and I. Eisen (see ASTM BULLETIN, *No. 151, March, 1948, p. 84 (TP 92). Discussion, No. 154 October, 1948, p. 44 (TP 202))*

A Visual Rating System for Rusted Steel Specimens—Harry L. Faigen (see ASTM BULLETIN, *No. 154, October, 1948, p. 39 (TP 197))*

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys—R. B. Smith (see ASTM BULLETIN, *No. 151, March 1948, p. 50 (TP 58))*

What's in an Alloy?—J. C. Kiszka (see ASTM BULLETIN, *No. 151, March, 1948, p. 51 (TP 59))*

Dynamic Calibration Method Uses Modified Proving Ring—R. C. A. Thurston (see ASTM BULLETIN, *No. 154, October, 1948, p. 50 (TP 208))*

The Action of "Chromic Acid" on Zinc Coatings—D. J. Swaine (see ASTM BULLETIN, *No. 154, October, 1948, p. 52. (TP 210) Discussion, No. 156, January, 1949, p. 49 (TP 11))*

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending—Harry Majors, Jr. (see ASTM BULLETIN, *No. 155, December, 1948, p. 39 (TP 247))*

Symposium on Usefulness and Limitations of Samples

Introduction—H. F. Dodge 857

Sampling and Its Uncertainties—S. S. Wilks 859

Discussion 876

Variation in Materials, Testing, and Sample Sizes—Leslie E. Simon 877

Discussion 882

The Amount of Inspection as a Function of Control of Quality—G. R. Gause 886

Discussion 894

Symposium on Deformation of Metals as Related to Forming and Service

PAGE
Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service (Special Technical Publication No. 87) ... 896

Panel Discussion on the Influence of Non-Ferrous Metals and Their Compounds on the Corrosion of Pressure Vessels

Station Design and Composition of Materials as Factors in Boiler Corrosion—R. B. Donworth ... 897
Discussion ... 905
Corrosion of High-Pressure Steam Generators: Status of Our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey ... 907
Discussion ... 926
Combined Bibliography ... 940

Symposium on Metallography in Color

Summary of Proceedings of the Symposium on Metallography in Color (Special Technical Publication No. 86) ... 942

Symposium on Magnetic Testing

Summary of Proceedings of the Symposium on Magnetic Testing (Special Technical Publication No. 85) ... 943

Cement, Concrete, Aggregates, Masonry Materials

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey and F. W. Barley ... 944
Discussion ... 959
New Type Weighted Needles for Determining the Setting Times of Magnesium Oxychloride Cements—Harry E. Chisolm (see ASTM BULLETIN, *No. 154, October 1948, p. 37 (TP 195)).*
Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—Walter H. Price and John W. Robison ... 962
Discussion ... 968
The Effect of Repeated Loading on the Bond Strength of Concrete—Supplement II—C. W. Muhlenbruch ... 977
Discussion ... 986
Research on Concrete Durability as Affected by Coarse Aggregate—Harold S. Sweet ... 988
Discussion ... 1017
Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor ... 1020
Discussion ... 1040
Testing Surface Waterproofers—F. O. Anderegg (see ASTM BULLETIN, *No. 156, January, 1949, p. 71 (TP 33)).*
Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests—Delmar L. Bloem (see ASTM BULLETIN, *No. 152, May, 1948, p. 76 (TP 128). Discussion, No. 153, August, 1948, p. 81 (TP 173)).*
An Instrument for Determining the Volume and Bulk Density of Granular Materials—Wm. E. Gross and John C. Goshorn (see ASTM BULLETIN, *No. 151, March, 1948, p. 55 (TP 63))*
Determination of the Air Content of Mortars by the Pressure Method—Thomas G. Taylor (see ASTM BULLETIN, *No. 155, December, 1948, p. 44 (TP 252))*

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete

Introduction ... 1055
Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton ... 1057
Discussion ... 1063
Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper ... 1067

PAGE

Tests Used by Bureau of Reclamation for Identifying Reactive Concrete Aggregates—R. C. Mielenz and L. P. Witte ... 1071
  Discussion ... 1104
A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and T. R. Smith ... 1108
  Discussion ... 1115
Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather ... 1120
  Discussion ... 1126

Symposium on Mineral Aggregates

Summary of Proceedings of the Symposium on Mineral Aggregates (Special Technical Publication No. 83) ... 1128

Symposium on Speed of Testing

Introduction—P. G. McVetty ... 1129
Testing Speed Limitations for Committee A-1 Specifications for Steel—Lawford H. Fry ... 1130
The Effect of Speed of Testing on Magnesium-Base Alloys—A. A. Moore ... 1133
Speed of Testing Wood: Factors in Its Control and Its Effect on Strength—L. J. Markwardt and J. A. Liska ... 1139
Effect of Speed of Test Upon Strength Properties of Plastics—Albert G. H. Dietz, W. J. Gailus, and S. Yurenka ... 1160
  Discussion ... 1187
Methods and Equipment for Controlling Speed of Testing—Lawrence K. Hyde ... 1191
  Discussion ... 1200

Petroleum Products and Lubricants

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab ... 1201
  Discussion ... 1211
Symposium on Cutting Oils: (see ASTM BULLETIN, *No. 151, March 1948)*
  Water-Mixed Cutting Fluids—Wm. H. Oldacre, (p. 45 (TP 53))
  Cutting Fluids—E. A. Reehl, (p. 47 (TP 55))
  Correlation of Laboratory Testing of Cutting Fluids with Field Tests—D. J. Wangelin, (p. 48 (TP 56))
Symposium on Turbine Oils: (see ASTM BULLETIN, *No. 152, May, 1948)*
  An Evaluation of Inhibited Turbine Oils Versus Uninhibited Turbine Oils for Turbine Lubrication—M. D. Baker p. 59 (TP 111).
  Service Experience with Turbine Oils—C. L. Pope and O. V. Sprague p. 62(TP 114).
  The Care of the Lubricant and Maintenance of the Lubricating System for Central Station Turbine Equipment—Ernest F. Walsh p. 64 (TP 116).
  Steam Turbines and Their Lubrication—C. D. Wilson p. 66 (TP 118).
The Thixotropic Characteristics of Lubricating Oil Greases—L. W. McLennan and G. H. Smith (see ASTM BULLETIN, *No. 152, May, 1948, p. 71 (TP 123).*
A Worker Consistometer for Lubricating Greases—S. A. McKee and H. S. White (see ASTM BULLETIN, *No. 153, August, 1948, p. 90 (TP 182).*
Induction Period Calculator—E. L. Korb (see ASTM BULLETIN, *No. 153, August, 1948, p. 99 (TP 191).*
The Light-Weight Cone for Penetration of Soft Greases—L. C. Brunstrum (see ASTM BULLETIN, *No. 154, October, 1948, p. 66 (TP 224).*
Requirements of an Engine Antifreeze and Methods of Evaluation—D. H. Green, J. C. Kratzer, and P. I. Emch (see ASTM BULLETIN, *No. 154, October, 1948, p. 57 (TP 215).*
Better Lead Scavenging Needed for Aviation Gasoline—E. A. Droegemueller (see ASTM BULLETIN, *No. 154, October 1948, p. 53 (TP 211).*
Photoelectric Measurement of a Color Index of Liquid Petroleum Products—Louis Lykken (see ASTM BULLETIN, *No. 154, October, 1948, p. 68 (TP 226).*

Symposium on Functional Tests for Ball-Bearing Greases

Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Greases (Special Technical Publication No. 84) ... 1214

Forum on Tractor Fuels

PAGE
Summary of Proceedings of the Forum on Tractor Fuels (Special Technical Publication No. 82) 1215

Petroleum Products as Related to Automotive Equipment

Summary of Proceedings of Petroleum Products as Related to Automotive Equipment (Special Technical Publication No. 81) 1216

Plastics

The Effect of Temperature on The Creep of Two Laminated Plastics *As Interpreted By The Hyperbolic-Sine Law and Activation Energy Theory*—W. N. Findley, W. J. Worley, and C. N. Adams........ 1217
A New Interpretation of Heat Distortion Data of Plastics—L. S. Moody and K. B. Goldblum (see ASTM BULLETIN, *No. 150, January, 1948, p. 55 (TP 27)).*
Tensile Stress-Strain Relationships of Laminated Plastics for Small Strains—J. J. Lamb and B. M. Axilrod (see ASTM BULLETIN, *No. 151, March, 1948, p. 59 (TP 67)).*
The Use of Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C.—F. E. Piech and W. E. Gloor (see ASTM BULLETIN, *No. 151, March, 1948, p. 70 (TP 278))*
Plastics as Optical Materials—H. R. Moulton (see ASTM BULLETIN, *No. 151, March, 1948, p. 75 (TP 83)).*
Low-Pressure Moldings and Laminates—T. Walter Noble (see ASTM BULLETIN, *No. 151, March, 1948, p. 77 (TP85)).*
Some Factors Affecting Adhesive Bond Formation—H. S. Meissner and E. W. Merrill (see ASTM BULLETIN, *No. 151, March, 1948, p. 80 (TP 88)).*
Metals and Plastics—Robert G. Chollar (see ASTM BUTTETIN, *No. 152, May, 1948, p. 80(TP 132)).*
The Effect of Fungi and Humidity on Plastics—John Leutritz, Jr. (see ASTM BULLETIN, *No. 152, May, 1948, p. 88 (TP 140)).*
The Mechanism of Plasticization in Plastics—J. Kenneth Craver, (see ASTM BULLETIN, *No. 152, May, 1948, p. 90 (TP 142)).*
Electrical Properties of Plastics—A. J. Warner (see ASTM BULLETIN, *No. 153, August, 1948, p. 60 (TP 152)).*
Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan (see ASTM BULLETIN, *No. 153, August, 1948, p. 65 (TP 157)).*
An Improved Low-Temperature Brittleness Test—E. F. Smith and G. J. Dienes (see ASTM BULLETIN, *No. 154, October, 1948, p. 46 (TP 204)).*

Paint

Mechanical Operation of the Bird Film Applicator—A. E. Jacobsen and H. S. Jensen (see ASTM BULLETIN, *No. 151, March, 1948, p. 95 (TP 103)).*
Measurement of Transparency of Surface Coatings—J. Gross, R. H. Kienle, L. A. Melsheimer, and E. I. Stearns (see ASTM BULLETIN, *No. 153, August, 1948, p. 73 (TP 165)).*
A Method for the Routine Determination of Dry Hiding Power of White Paints—F. C. Schmutz and J. M. Gallagher (see ASTM BULLETIN, *No. 153, August, 1948, p. 77 (TP 169)).*
The Gloss Measurement of Paint Finishes—Richard S. Hunter (see ASTM BULLETIN, *No. 150, January, 1948, p. 72 (TP 11)).*

Textiles

Symposium on Flammability (see ASTM BULLETIN, *No. 150, January, 1948)*
  Flammability of Textiles for Garment Purposes—Frederic Bonnet p. 29 (TP 1).
  Flameproofing in Theory and Practice—R. W. Little p. 34 (TP 6).
  The Retailer's Viewpoint on the Flammability of Textiles—Charles W. Dorn p. 36 (TP 8).
Measurement and Elimination of Inertia Effects in Textile Testing Equipment by Means of Electric Strain Gages—Herbert F. Schiefer (see ASTM BULLETIN, *No. 150, January, 1948, p. 43 (TB 13))*
A Stress-Strain Tester for Textiles Employing a Magnetic Strain Gage—J. W. Ballou (see ASTM BULLETIN, *No. 150, January, 1948, p. 38 (TB 10))*

PAGE

Service Wearing Tests—E. N. Ditton (see ASTM BULLETIN, *No. 152, May, 1948, p. 93 (TP 145))*

Report of the Task Group Studying the Wyzenbeek Precision Wear Test Meter—S. J. Tanenhaus and Gerald Winston (see ASTM BULLETIN, *No. 154, October, 1948, p. 74 (TP 232))*

The Importance of the Time Factor in the Study of Textile Properties—E. R. Schwartz (see ASTM Standards on Textile Materials, 1948, p. 485)

Some Applications of Stress-Strain Measurements to the Determination of Fiber Properties—A. M. Sookne (see ASTM Standards on Textile Materials, 1948, p. 498)

The Stress-Strain Diagram as a Tool in Textile Research—W. J. Hamburger (see ASTM Standards on Textile Materials, 1948, p. 491)

Miscellaneous

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams—W. G. Holt **1240**

The Importance and Practical Use of Relative Density in Soil Mechanics—D. M. Burmister **1249**

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method—J. Jonakin, P. Cohen, R. Corey, and B. Jain **1269**

Discussion **1290**

Nontoxic Rubber for Gas Masks—S. H. Katz **1293**

Discussion **1299**

Measurement of Volume Resistivity of Electrically Conducting Rubber-Like Material—R. F. Miller (see ASTM BULLETIN, *No. 151, March, 1948, p. 91 (TP 99))*

Some Fundamentals of Glass Adhesion—Frank Moser (see ASTM BULLETIN, *No. 150, January, 1948, p. 51 (TP 23)).*

Fungus Resistance of Treated Vegetable Fiber Gaskets—Sigmund Berk (see ASTM BULLETIN, *No. 150, January, 1948, p. 46 (TP 18)).*

Load Concentration in Corrugated Paper—J. H. Toulouse and P. J. Barcus (see ASTM BULLETIN, *No. 152, May, 1948, p. 77 (TP 129)).*

A Means of Determining the Hardness of Wood and Modified Woods over a Broad Specific Gravity Range—Richard C. Weatherwax, E. C. O. Erickson, and Alfred J. Stamm (see ASTM BULLETIN, *No. 153, August, 1948, p. 84 (TP 176))*

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell (see ASTM BULLETIN, *No. 151, March, 1948, p. 88 (TP 96))*

A Method of Calibrating Extensometers—W. C. Aber and F. M. Howell (see ASTM BULLETIN, *No. 155, December, 1948, p. 33 (TP 241)).*

One-Millionth Second Radiography and Its Development—Charles M. Slack and Louis F. Ehrke (see ASTM BULLETIN, *No. 150, January, 1948, p. 59 (TP 31)).*

Frankford Arsenal Experience with High-Speed Radiography—E. R. Thilo (see ASTM BULLETIN, *No. 150, January, 1948, p. 69 (TP 41)).*

Research—Everybody's Doing It Now—T. A. Boyd (see ASTM BULLETIN, *No. 152, May, 1948, p. 53 (TP 105)).*

The Use of a Test Track in Developing Good Packaging and Carloading Methods—A. N. Perry (see ASTM BULLETIN, *No. 155, December, 1948, p. 38 (TP 246)).*

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers—C. J. Seiler and A. D. McLaren (see ASTM BULLETIN, *No. 155, December, 1948, p. 50 (TP 258)).*

The Preparation of Uniform Films and Their Dependence on the Irregularities of the Base Surface—Stig Johanson (see ASTM BULLETIN, *No. 155, December, 1948, p. 53 (TP 261)).*

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts—W. W. Buechner, R. J. Van de Graaff, H. Feshbach, E. A. Burrill, A. Sperduto, and L. R. McIntosh (see ASTM BULLETIN, *No. 155, December, 1948, p. 54 (TP 262)).*

Subject Index **1300**

Author Index **1335**

List of Special Technical Publications **1352**

SUMMARY OF PROCEEDINGS

OF THE

FIFTY-FIRST ANNUAL MEETING

DETROIT, MICH., JUNE 21–25, 1948

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards appearing in the annual reports of the technical committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentatives are published in the 1948 Supplement to Book of A.S.T.M. Standards, Parts I-A and I-B, II, and III-A and III-B. Except as noted, the reports and papers appear in this volume.

This Summary of the Fifty-first Annual Meeting of the American Society for Testing Materials, held in Detroit, Mich., June 21–25, 1948, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. The A.S.T.M. Headquarters and Exhibit were in the Book-Cadillac Hotel with committee meetings and sessions also being held at the Statler and Detroit-Leland Hotels. This Summary indicates which sessions were held at other than the Headquarters Hotel. In all, 19 technical sessions were held, as well as the Eighth Exhibit of Testing Apparatus and Related Equipment, and the Sixth A.S.T.M. Photographic Exhibit. The exhibit of testing apparatus and the photographic exhibit are described in some detail in the August ASTM BULLETIN.

The registered attendance of the meeting is as follows: Members present or represented, 1160; committee members, 358; guests, 250; total, 1768; ladies, 133.

The Proceedings are set forth session by session. There were 57 reports and 95 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all the reports together with the page reference where the action thereon is recorded:

---

[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 30, 1948, and all actions were approved.

Location of Actions on Committee Reports

PAGE

Committee:
- Board of Directors 4
- A-1 on Steel 11
- A-3 on Cast Iron 12
- A-5 on Corrosion of Iron and Steel 6
- A-6 on Magnetic Properties 5
- A-7 on Malleable-Iron Castings 12
- A-9 on Ferro-Alloys 12
- A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys 7

- B-1 on Wire for Electrical Conductors 18
- B-2 on Non-Ferrous Metals and Alloys 19
- B-3 on Corrosion of Non-Ferrous Metals and Alloys 18
- B-4 on Electrical-Heating, Resistance and Related Alloys 19
- B-5 on Copper and Copper Alloys, Cast and Wrought 19
- B-6 on Die-Cast Metals and Alloys 20
- B-7 on Light Metals and Alloys, Cast and Wrought 20
- B-8 on Electrodeposited Metallic Coatings 18
- B-9 on Metal Powders and Metal Powder Products 21

- C-1 on Cement 21
- C-2 on Magnesium Oxychloride Cements 9
- C-4 on Clay Pipe 9
- C-7 on Lime 9
- C-8 on Refractories 9
- C-9 on Concrete and Concrete Aggregates 21
- C-11 on Gypsum 22
- C-12 on Mortars for Unit Masonry 9
- C-14 on Glass and Glass Products 9
- C-16 on Thermal Insulating Materials 22
- C-18 on Natural Building Stone 9

- D-1 on Paint, Varnish, Lacquer, and Related Products 10
- D-2 on Petroleum Products and Lubricants 7
- D-3 on Gaseous Fuels 17
- D-4 on Road and Paving Materials 12
- D-5 on Coal and Coke 17
- D-6 on Paper and Paper Products 17
- D-7 on Wood 17
- D-8 on Bituminous Waterproofing and Roofing Materials 8
- D-9 on Electrical Insulating Materials 24
- D-10 on Shipping Containers 17
- D-11 on Rubber and Rubber-Like Materials 23
- D-12 on Soaps and Other Detergents 17
- D-13 on Textile Materials 14
- D-14 on Adhesives 24
- D-15 on Engine Antifreezes 23
- D-17 on Naval Stores 24
- D-18 on Soils for Engineering Purposes 8
- D-19 on Water for Industrial Uses 13
- D-20 on Plastics 23

- E-1 on Methods of Testing 14
- E-2 on Spectrographic Analysis 14
- E-3 on Chemical Analysis of Metals 21
- E-4 on Metallography 4
- E-5 on Fire Tests of Materials and Construction 18
- E-6 on Methods of Testing Building Construction 18
- E-7 on Radiographic Testing 4
- E-8 on Nomenclature and Definitions 14
- E-9 on Fatigue 12
- E-11 on Quality Control of Materials 4

Joint Research Committee on Filler Metal 12
Joint Research Committee on Effect of Temperature on the Properties of Metals 6
Joint Research Committee on Boiler Feedwater Studies 14

SYMPOSIUMS

PAGE
Round-Table Discussion on Ultra-Sonic Testing 3
Symposium on Metallography in Color 4
Symposium on Usefulness and Limitations of Samples 4
Symposium on Magnetic Testing 5
Symposium on Deformation of Metals as Related to Forming and Service 6
Symposium on Functional Tests for Ball-Bearing Greases 8
Symposium on Mineral Aggregates 12
Panel Discussion on Corrosion of Pressure Vessels 13
Symposium on Speed of Testing 15
Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete 15

ROUND-TABLE DISCUSSION ON ULTRA-SONIC TESTING

MONDAY, JUNE 21, 2 P.M.

CHAIRMAN: L. W. BALL

This Round Table Discussion was sponsored by Committee E-7 on Radiographic Testing in view of its proposed expansion of scope to include nondestructive testing in general. The field of ultra-sonic testing is becoming increasingly important commercially and this discussion was arranged to obtain an interchange of information among persons in the field.

FIRST SESSION—OPENING SESSION; SYMPOSIUM ON METALLOGRAPHY IN COLOR

Opening Session

MONDAY, JUNE 21, 8 P.M.

SESSION CHAIRMAN: PRESIDENT T. A. BOYD

In opening the Fifty-first Annual Meeting of the Society, President Boyd recalled that this was the second annual meeting held in Detroit, the previous one being in 1935. He further pointed out that at that time it was possible to hold the entire meeting in one hotel, whereas the facilities of three hotels are being used for this annual meeting. He called attention to the very complete technical program for the meeting which consisted of 19 sessions and, in addition, some 375 meetings of committees and subcommittees. He also directed attention to the Eighth Exhibit of Testing Apparatus and Related Equipment and to the Sixth Photographic Exhibit which were important features of the meeting.

President Boyd also expressed appreciation to the General Committee on Arrangements, which functioned under the auspices of the A.S.T.M. Detroit District Council, for the large part which it played in making arrangements for the meeting, and in particular for the ladies entertainment and for the annual dinner and dance to be held on Wednesday evening.

Minutes of Fiftieth Annual Meeting

The minutes of the Fiftieth Annual Meeting were distributed the during summer of 1947 as a Summary of Proceedings. This summary is published in the 1947 *Proceedings*.[2] On motion, the minutes were approved.

---

[2] Summary of Proceedings of the Fiftieth Annual Meeting, *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 1 (1947).

Report of Board of Directors

In presenting the Board of Directors Report, the Executive Secretary, C. L. Warwick, called attention first to the reports of the following Administrative Committees that were appended to the report:

- Administrative Committee on Standards
- Administrative Committee on Research
- Administrative Committee on Papers and Publications
- Administrative Committee on Ultimate Consumer Goods
- Administrative Committee on Simulated Service Testing
- Administrative Committee on District Activities

These brief reports summarized the activities in the designated fields of work.

Mr. Warwick stressed the great activity by the members and Society committees in both standardization and research on engineering materials. He also reviewed a number of important aspects of the Society's work, including membership; meetings, both national and local; headquarters; publications; technical activities; organization of new committees; relations with other organizations; cooperation with the American Standards Association; and consideration of international standardization activities.

The finances of the Society were reviewed at length, including the presentation of the auditors report for the previous fiscal year.

Committee E-7 on Radiographic Testing:

Oral progress report presented by H. H. Lester, chairman.

Committee E-4 on Metallography:

Report presented from manuscript by the chairman, L. L. Wyman.

Symposium on Metallography in Color[3]

MONDAY, JUNE 21, 8:30 P.M.

SYMPOSIUM SESSION CHAIRMAN: L. V. FOSTER

The Symposium on Metallography in Color consisted of the following five papers:

- Microscope Optics for Color Metallography—J. R. Benford, presented from manuscript by the author.
- Quality and Quantity of Illumination in Metallography—R. P. Loveland, presented from manuscript by the author.
- Color Metallography Simplified—Mason Clogg, Jr., presented from manuscript by Mr. Gordon Bedford.
- Some Applications of Color Metallography—W. D. Forgeng, presented from manuscript by the author.
- The Application of Color Photography to the Study of Nonmetallic Inclusions—A. M. Hall and E. E. Fletcher, presented from manuscript by Mr. Fletcher.

SECOND SESSION—SYMPOSIUM ON USEFULNESS AND LIMITATIONS OF SAMPLES

TUESDAY, JUNE 22, 9:30 A.M.

DETROIT-LELAND HOTEL

SESSION CO-CHAIRMEN: PAST-PRESIDENT ARTHUR W. CARPENTER; H. F. DODGE

SECRETARY: O. P. BECKWITH

Committee E-11 on Quality Control of Materials:

Report presented by H. F. Dodge, chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Rec. Practices for Designation of Numerical Requirements in Standards (E 29 - 40 T)

Symposium on Usefulness and Limitations of Samples:

The Symposium on Usefulness and

---

[3] To be issued as a separate publication.

Limitations of Samples consisted of the following three papers:

Sampling and Its Uncertainties—S. S. Wilks, presented from manuscript by the author.

Variation in Materials, Testing, and Sample Sizes—Leslie E. Simon, presented by the author.

The Amount of Inspection as a Function of Control of Quality—G. R. Gause, presented from manuscript by the author.

THIRD AND FOURTH SESSIONS—SYMPOSIUM ON MAGNETIC TESTING[3]

Third Session

TUESDAY, JUNE 22, 9:30 A.M.

SESSION CHAIRMAN: R. L. SANFORD

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following actions taken:

Adopted as Standard:

Test for Permeability of Paramagnetic Materials (A 34 – 47 T)

Adopted as Standard, Revisions in:

Methods of Testing Magnetic Materials (A 34 – 47)

Definitions of Terms, with Symbols, Relating to Magnetic Testing (A 127 – 46)

Symposium on Magnetic Testing:[3]

The Symposium on Magnetic Testing consisted of the following ten papers as presented in the third and fourth sessions:

Testing Magnetic Materials—B. M. Smith, presented from manuscript by the author.

Permanent Magnet Testing Methods and Their Validity in Determining Product Performance—C. A. Maynard and J. E. Mitch, presented from manuscript by Mr. Maynard.

Core Loss Test for Narrow Silicon Steel Strip—J. A. Ashworth, presented from manuscript by the author.

D-C Permeability Testing of Epstein Samples with Double - Lapped Joints — D. C. Dieterly, presented from manuscript by the author.

A New D-C Permeameter—W. J. Carr, Jr., presented from manuscript by the author.

Fourth Session

TUESDAY, JUNE 22, 2 P.M.

SESSION CHAIRMAN: THOMAS SPOONER

Variation of Core Loss and Permeability of Electrical Grade Silicon Sheet Steel—A. C. Beiler and P. L. Schmidt, presented from manuscript by Mr. Beiler.

The Evaluation of Hysteresis Core Loss by Power Equations—Horatio W. Lamson, presented from manuscript by the author.

Magnetic Analysis Inspection in the Steel Industry—Theodore Zuschlag, presented from manuscript by the author.

Magnetic Stress Analysis—P. E. Cavanagh and T. Wlodek, presented from manuscript by Mr. Cavanagh.

The Testing of Magnetic Recording Media—D. E. Wiegand, presented by the author.

MOVIE ON MODERN QUALITY CONTROL

PRECEDED BY A TALK BY SIMON COLLIER, JOHNS-MANVILLE, CORP.

TUESDAY, JUNE 22, 4:30 P.M.

CHAIRMAN: PAST-PRESIDENT ARTHUR W. CARPENTER

As part of an extensive training program at Johns-Manville Corp. designed to teach the most modern techniques of Quality Control to their production and inspection personnel, a very interesting sound color film was developed. The film illustrated the various types of rejects and their causes and the advantages of statistical quality control as applied specifically to methods of sampling.

Mr. Collier, Director of Quality, Johns-Manville Corp., delivered a very interesting and instructive talk describing the background of the preparation of this film and then had the film shown.

An audience of over 300 received this excellent presentation with much enthusiasm.

FIFTH SESSION—SYMPOSIUM ON DEFORMATION OF METALS AS RELATED TO FORMING AND SERVICE[3]

TUESDAY, JUNE 22, 8 P.M.

SESSION CHAIRMAN: L. L. WYMAN

Tests of Ductility in Ship Structure—W. P. Roop, presented from manuscript by the author.

Notch Bar Tension Tests on Annealed Carbon Steel Specimens of Various Sizes and Contours—G. Sachs and M. L. Fried, presented from manuscript by Mr. Sachs.

Notch-Sensitivity in Ship-Plate Correlation of Laboratory Scale Tests with Large Scale Plate Tests—Noah A. Kahn and E. A. Imbembo, presented from manuscript by Mr. Kahn.

Measurement of Ductility in Sheet Metals—John R. Low, Jr., and Thomas A. Prater, presented from manuscript by Mr. Prater.

Hydraulic Bulge Testing of Sheet Metals—W. Lankford, presented from manuscript by the author.

SIXTH SESSION—EFFECT OF TEMPERATURE ON PROPERTIES OF METALS

WEDNESDAY, JUNE 23, 10 A. M.

DETROIT-LELAND HOTEL

SESSION CHAIRMAN: H. C. CROSS

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Report presented from manuscript by N. L. Mochel, chairman.

Appendices:

Studies on Graphitization Susceptibility of Carbon-Molybdenum and Chromium-Molybdenum Steels (Project No. 29)—J. J. Kanter and E. A. Sticha, presented from manuscript by Mr. Sticha.

High Temperature Bolting Materials (Project No. 16)—Ernest L. Robinson, presented by the author.

Papers:

The following papers were presented:

High-Temperature Properties of Rotar Disks for Gas Turbines as Affected by Variables in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, presented from manuscript by Mr. Freeman.

Fatigue and Static Load Tests of an Austenitic Cast Iron at Elevated Temperatures—W. Leighton Collins, presented from manuscript by the author.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—Paul G. Jones and W. J. Worley, presented by Mr. Jones.

SEVENTH SESSION—CORROSION OF FERROUS METALS

WEDNESDAY, JUNE 23, 2 P.M.

DETROIT-LELAND HOTEL

SESSION CHAIRMAN: T. R. GALLOWAY

Committee A-5 on Corrosion of Iron and Steel:

Report presented by H. F. Hormann, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 46), except the proposed revision in Section 17 which was withdrawn by the committee.

Adopted as Standard, Revisions in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 39), with the following information covering the No. 14½ Farm Fencing added to the new Table I:

Design Numbers	Number of Horizontal Wires	Height, in.	Spacing of Stay Wires, in.	Intermediate Line and Stay Wires	Top and Bottom Wires
No. 14½ FARM					
939-6-14½	9	39	6	14½	11
832-6-14½	8	32	6	14½	11
726-6-14½	7	26	6	14½	11

Spec. for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 39), with the table in Section 2 further revised to read as follows:

Size, Steel Wire Gage	Nominal Diameter of Zinc-Coated Wire in Strand, in.	Number of Barbs	Barb Spacing, in.	Diameter of Barbs, Steel Wire Gage	Shape of Barbs
No. 12½	0.099	2	4	14	round
No. 12½	0.099	4	5	14[a]	half-round
No. 12½	0.099	4	5	14	round
No. 12½	0.099	2	4	12½[a]	flat
No. 14	0.080	2	4	16	round

[a] The gage of the half-round and flat barbs is the same as that of the full-round wire section from which the barbs are made.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented from manuscript by Jerome Strauss, chairman.

Papers:

The following papers were presented:

Quantitative Evaluation of Corrosive Conditions—W. F. Bonwitt and I. Eisen, presented by Mr. Bonwitt.[4]

Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, presented by the author.

Laboratory Corrosion Tests of Iron and Stee Pipes—George A. Ellinger, Sybil B. Marzolf, and Leo J. Waldron, presented by Mr. Ellinger.

An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion Fatigue Failures in Steel—Robert C. McMaster, presented from manuscript by the author.

Atmospheric Durability of Steel Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, presented by Mr. H. R. Copson.

EIGHTH SESSION—SYMPOSIUM ON FUNCTIONAL TESTS FOR BALL-BEARING GREASES[3]

WEDNESDAY, JUNE 23, 2 P.M.

SESSION CHAIRMAN: R. C. ADAMS

Committee D-2 on Petroleum Products and Lubricants:

Report presented by C. Dantsizen, chairman, and the following actions taken:

Accepted as Tentative:

Test for Interfacial Tension of Oil Against Water by the Ring Method

Test for Evaporation Loss of Lubricating Greases and Oils

Test for Determination of Butadiene Content of Polymerization Grade Butadiene

Test for Neutralization Value (Acid and Base Numbers) by Color Indicator Method

Test for Color of U. S. Army Motor Fuel (All-Purpose) by Means of A.S.T.M. Color Standard (former Emergency Method ES-32)

Spec. for Diesel Fuel Oils

Accepted as Tentative, Revisions in:

Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 – 46 T)

Spec. for Gasoline (D 439 – 40 T)

Spec. for Aviation Gasolines (D 910 – 47 T)

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 – 43)

Test for Tetraethyllead in Gasoline (D 526 – 42)

Adopted as Standard:

Test for Cone Penetration of Lubricating Grease (D 217 – 47 T), as revised

Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils (D 810 – 47 T)

Chemical Analysis for Metals in Lubricating Oils (D 811 – 44 T)

Test for Sediment in Fuel Oil by Extraction (D 473 – 46 T), as revised

Adopted as Standard, Revisions in:

Definitions of Terms Relating to Petroleum (D 288 – 47)

---

[4] Published in the ASTM BULLETIN, No. 151, March, 1948, p. 84.

Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 47), comprising the following changes:[4a]

(*1*) Inclusion as optional equipment of the Detonation Meter, as preprinted.

(*2*) Inclusion of the following revised Section 14 on Reproducibility:

14. *Reproducibility of Results.*—Extensive data from independent laboratories over a number of years for many samples of conventional motor gasolines have shown a standard deviation of 0.5 octane number (an average deviation of about 0.4 octane number). Based on this standard deviation, the number of tests required to yield a rating of desired accuracy is given in the following table:

Accuracy Desired, Octane Number, plus or minus	Number of Tests Required to Obtain the Desired Accuracy		
	9 times out of 10	19 times out of 20	99 times out of 100
1.0......	1	1	2
0.5......	2	3	5
0.3......	6	9	15

This table shows that a single rating can normally be expected to be within ±1.0 octane number of the true value in about 19 cases out of 20. On the other hand, if an accuracy of ±0.3 octane number is desired with a certainty of 90 per cent (9 times out of 10) it is necessary to obtain an average of six ratings in six different engines (preferably in six independent laboratories).

Although the above degree of reproducibility applies to conventional motor gasolines when careful attention is given to the details of test procedure and engine condition, it does not necessarily apply to fuels which differ materially from finished motor gasolines. In such cases, the reproducibility is likely to be lower and result in higher standard deviations.

Withdrawal of Tentative:

Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration (D 663 - 46 T)

Editorial Change Accepted in:

Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (D 664 - 46 T)

Accepted for Publication as Information Only:

Method of Test for Performance Characteristics of Wheel Bearing Grease

Method of Test for Separation of Residue from Butadiene

Method of Test for Butadiene Dimer in Polymerization Grade Butadiene

Method of Test for Nonvolatile Residue of Polymerization Grade Butadiene

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion

Paper:

Bearing Corrosion Tests Using the MacCoull Tester and a Study of the Kinetics of Oil Oxidation—Hugh R. Lehman and L. Kermit Herndon, presented from manuscript by Mr. Herndon. [Withheld from publication in 1948.—ED.]

Symposium on Functional Tests for Ball-Bearing Greases:[3]

Development of Functional Grease Test Methods for the Aircraft Industry—D. H. Moreton, presented from manuscript by the author.

Grease—An Oil Storehouse for Bearings—D. F. Wilcock and Marshall Anderson, presented from manuscript by Mr. Wilcock.

Laboratory Performance Tests for Anti-Friction Bearing Greases—M. Herbst, W. A. Prendergast, R. S. Barnett, W. J. Finn, and O. P. Puryear, presented from manuscript by Mr. Barnett.

Service Experience with Grease—C. L. Pope and W. T. Everitt, presented from manuscript by Mr. Pope.

Factors Affecting Simulated Service Tests of Greases—Sidney M. Collegeman and John R. Belt, presented from manuscript by Mr. Collegeman.

NINTH SESSION—WATERPROOFING, BRICK, MASONRY MATERIALS

WEDNESDAY, JUNE 23, 2 P.M.

SESSION CHAIRMAN: D. E. PARSONS

Committee D-18 on Soils for Engineering Purposes:

Report presented from manuscript by W. S. Housel, first vice-chairman.

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented from manuscript by E. H. Berger, vice-chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Methods of Sampling Bituminous Materials

[4a] This recommendation, not preprinted, was accepted by unanimous consent, subject to unanimous affirmative letter ballot of Committee D-2, which ballot has been favorable.

(D 140 - 46 T), jointly with Committee D-4 on Road and Paving Materials.

Committee C-2 on Magnesium Oxychloride Cements:

Report presented from manuscript by G. J. Fink, secretary.

Committee C-4 on Clay Pipe:

Report presented from manuscript, in the absence of the chairman, by L. C. Gilbert.

Committee C-7 on Lime:

Report presented from manuscript by W. C. Voss, chairman.

Committee C-8 on Refractories:

Report presented by J. D. Sullivan, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Definitions of Terms Relating to Refractories (C 71 - 47)
Method of Panel Spalling Test for Fireclay Plastic Refractories (C 180 - 47)

Adopted as Standard, Revisions in:

Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 47)
Spec. for Refractories for Incinerators (C 106 - 47)
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 47)

Committee C-12 on Mortars for Unit Masonry:

Report presented from manuscript by H. C. Plummer, secretary.

It was announced that proposed Tentative Specifications for Mortar for Unit Masonry would be presented to the Society subsequent to the meeting through the Administrative Committee on Standards.

Committee C-14 on Glass and Glass Products:

Report presented from manuscript, in the absence of the chairman, by L. C. Gilbert.

Committee C-18 on Natural Building Stones:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Accepted as Tentative:

Test for Durability of Slate for Roofing
Test for Combined Effect of Temperature Cycles and Weak Salt Solutions
Definitions of Terms Relating to Natural Building Stones

Adopted as Standard, Revisions in:

Definition of the Term Slate (C 119 - 27)
Test for Water Absorption of Slate (C 121 - 31)
Methods of Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (C 120 - 31)

Papers:

New Type Weighted Needles for Determining the Setting Times of Magnesium Oxychloride Cements—Harry E. Chisholm, presented from manuscript by Mr. W. J. Riley.[5]
Testing Surface Waterproofers—F. O. Anderegg, presented by the author.[6]
Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, presented by the author.

ANNUAL DINNER AND DANCE

WEDNESDAY, JUNE 23, 6:30 P.M.

STATLER HOTEL

CHAIRMAN: C. H. FELLOWS

The Annual Meeting Dinner was attended by about 300 members and guests.

At the conclusion of the dinner, Mr. Fellows extended a very cordial welcome from the Detroit District Council which had served as host in making arrangements for this social event which highlighted the Fifty-first Annual Meeting of the Society.

Election of Officers:

The chairman then recognized H. A. Wagner, chairman of the Committee of Tellers, who reported the results of the letter ballot on election of officers. Of the 1157 ballots cast, the results were as follows:

---

[5] Published in ASTM BULLETIN, No. 154, October, 1948, p. 37.

[6] Published in ASTM BULLETIN, No. 156, January, 1949, p. 71.

For President, to serve for one year:
  R. L. Templin, 1157 votes.
For Vice-President, to serve for two years:
  L. J. Markwardt, 1157 votes.
For Directors, to serve for three years:
  L. C. Beard, Jr., 1157 votes.
  Simon Collier, 1157 votes.
  T. P. Dresser, Jr., 1157 votes.
  E. W. Fasig, 1157 votes.
  H. K. Nason, 1157 votes.

The chairman then introduced the newly elected members of the Board of Directors, requesting that they rise as as their names were called.

The chairman then presented the President-Elect, R. L. Templin, who expressed sincere appreciation for the honor which the election conferred upon him and said that he fully realized the duties and responsibilities which the office entailed, especially at this time with the extensive increase in activities of the Society.

Award of Honorary Memberships:

President Boyd then made awards of honorary memberships to the following three members of the Society:

Harvey L. Curtis
Frank H. Jackson
William B. Price

Prior to presentation, C. L. Warwick, Executive Secretary, reviewed briefly the accomplishments of each of the recipients and directed particular attention to the services or contributions that each had rendered to the Society.

The two recipients of honorary membership who were present, Mr. Curtis and Mr. Jackson, then in turn expressed appreciation of the honor conferred upon them.

Presidential Address:

The chairman then presented the President, T. A. Boyd, who delivered the presidential address entitled "If Doctor Marburg Came Back Today."[7]

The Annual Dinner was followed by dancing provided by the Detroit District Council, through its Committee on Arrangements.

TENTH SESSION—FATIGUE—FERROUS METALS; PAINT

THURSDAY, JUNE 24, 9:30 A.M.
DETROIT-LELAND HOTEL
SESSION CHAIRMAN: VICE-PRESIDENT J. G. MORROW

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Raw Castor Oil, with a change to use the 1933 Gardner standards for color requirement and addition of reference to Method D 154 for the color test.
Spec. for Dehydrated Castor Oil
Spec. for Heavy Mineral Spirits
Spec. for Secondary Butyl Acetate (85 to 88 per cent Grade)
Spec. for Aluminum Pigment, Powder and Paste for Paints, to replace the Standard Spec. for Aluminum Powder for Paints (Aluminum Bronze Powder) (D266 – 41), and for Aluminum Pigment Paste for Paint (D 474 – 41)
Spec. for Copper Phthalocyanine Blue
Spec. for Copper Powder
Method of Test for Heat Bodying Rate of Drying Oils
Method of Test for Abrasion Resistance of Organic Coatings by the Falling Sand Method
Method of Test for Evaluating Degree of Resistance of Traffic Paint to Bleeding
Method of Test for Para Red and Toluidine Red Pigments

Accepted as Tentative, Revisions in:

Spec. for Wood to Be Used as Panels in Weathering Tests of Paints and Varnishes (D 358 – 44 T)
Spec. for Red and Brown Iron Oxide Pigments (D 84 – 46 T)

---

[7] See p. 26.

Spec. for Venetian Red (D 767 - 44 T)
Methods of Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (D 283 - 47 T)
Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 47 T)
Test for Specular Gloss of Paint Finishes (D 523-44 T)
Spec. for C. P. Toluidine Toner (D 656-43)
Spec. for C. P. Para Red Toner (D 475-41), to replace the Standard Specifications for Reduced Para Red (D 264-47)
Method of Test for Acetone Extract in Dry Lampblack and Dry Bone Black (D 305-31)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16-47)

Adopted as Standard:

Spec. for Black Synthetic Iron Oxide (D 769-44 T)
Spec. for Pumice Pigment (D 867-46 T)
Method of Evaluating Degree of Resistance of Traffic Paint to Bleeding (D 868-46 T)
Test for Evaluating Degree of Settling of Traffic Paint (D 869-46 T)
Methods of Sampling and Testing Turpentine (D 233-44 T)
Methods of Testing Cellulose Acetate (D 871-46 T)
Spec. for Titanium Dioxide Pigments (D 476-46 T), as revised, replacing the Spec. for Titanium Dioxide Pigments (D 476-41)
Spec. for Raw Umber (D 763-44 T) and for Burnt Umber (D 764-44 T), as revised and combined into a single standard
Spec. for Raw Sienna (D 765-44 T) and for Burnt Sienna (D 766-44 T), as revised and combined into a single standard
Spec. for Orange Shellac and Other Lacs (D 237-43 T), as revised and with two corrections in Table I: under grade D change the orpiment requirement from "0.02" to read "0.2" per cent max.; under type III change the wax requirement from "5.5" to read "3.5" per cent max.
Test for Dry No-Pick-Up Time of Traffic Paint (D 711 - 46 T), as revised

Adopted as Standard, Revisions in:

Spec. for Raw Tung Oil (D 12-41)
Spec. for Raw Soybean Oil (D 124-41), except to omit the change in title
Spec. for Perilla Oil, Raw or Refined (D 125-47)
Spec. for Raw Linseed Oil (D 234-28)
Spec. for Boiled Linseed Oil (D 260-33)
Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268-46)
Spec. and Tests for Soluble Nitrocellulose (D 301-33)
Methods of Sampling and Testing Aluminum Powder and Paste (D 480-44)

Withdrawal of Standards:

Spec. for Reduced Para Red (D 264-47)
Spec. for Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266-41)
Spec. for Aluminum Pigment Paste for Paint (D 474-41)

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Method of Tension Testing of Steel Spring Wire
Method of Macroetch Testing and Inspection of Steel Forgings

Accepted as Tentative, Revisions in:

Spec. for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158-47 T)
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206-47 T)
Spec. for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280-47 T)
Spec. for Seamless 1 Per Cent Chromium, 0.5 Per Cent Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315-47 T)

In the above four specifications, the section on finish is to be revised as preprinted, plus the following additional paragraph:

(*2*) Pipe showing inside or outside surface checks (fish scale) $\frac{1}{64}$ in. or less in depth need not have these defects removed. Such defects over $\frac{1}{64}$ in. but not more than $\frac{1}{32}$ in. in depth shall be removed by machining or grinding. Pipe on which these defects are more than $\frac{1}{32}$ in. in depth shall be rejected, unless the manufacturer can demonstrate to the purchaser that the defects are not injurious as defined in Paragraph (*b*).

Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 47 T)
Spec. for Heavy Gage Structural Quality Flat Hot-Rolled Carbon-Steel Sheets (A 245 - 47 T)
Spec. for Light Gage Structural Quality Flat Hot-Rolled Carbon-Steel Sheets (A 246 - 47 T)
Spec. for Hot-Rolled Strip of Structural Quality (A 303 - 47 T)
Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182-46), except that grade F 8t will not be deleted from the revised specifications, also the designation F 2 will be assigned the new 1 per cent chromium, 0.5 per cent molybdenum grade.
Spec. for Electric Fusion Welded Steel Pipe (Sizes 4 in. to but not including 30 in.) (A 139-46)

Adopted as Standard, Revisions in:

Spec. for High-Carbon Steel Joint Bars (A 5-39), with a minor change in the revised Section 14 on Marking and Stamping, changing the words "section number" to read "section designation."

Spec. for Wrought Steel Wheels for Electric Railway Service (A 25-46)

Spec. for Quenched Carbon-Steel Joint Bars (A 49-39)

Spec. for Multiple-Wear Wrought Steel Wheels (A 57-46)

Spec. for Steel Tie Plates (A 67-46), with a minor revision in the new Section 12 on Marking, changing the words "The plan designation of the tie plate" to read "The tie plate section designation."

Spec. for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194-46)

Spec. for Hot-Worked High-Carbon Steel Tie Plates (A 241-41), with a minor revision in the new Section 11 on Marking, changing the words "The plan designation of the tie plate" to read "The tie plate section designation."

Editorial Changes Accepted in:

Spec. for Steel Helical Springs (A 61-39)

Spec. for Steel Elliptical Springs (A 62-39)

Spec. for Heat-Treated Steel Helical Springs (A 125-39)

Spec. for Heat-Treated Steel Elliptical Springs (A 147-30)

Committee A-9 on Ferro-Alloys:

Report presented from manuscript by W. C. Bowden, Jr., chairman.

Joint Committee on Filler Metal:

Report presented from manuscript, in the absence of the chairman, by C. S. Cole.

Committee E-9 on Fatigue:

Report presented from manuscript by R. E. Peterson, chairman.

Committee A-3 on Cast Iron:

Report presented by J. T. MacKenzie, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Gray Iron Castings for Non-Pressure Containing Parts at Elevated Temperatures

Adopted as Standard, Revisions in:

Spec. for Gray Iron Castings (A 48-46)

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Pearlitic Malleable Iron Castings (A 220-47 T), with a further change adding the words "or period" at the end of the new Section 4 (*c*).

Adopted as Standard, Revisions in:

Spec. for Malleable Iron Castings (A 47-47), with a further change adding the words "or period" at the end of the new Section 5 (*c*).

Papers:

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, presented from manuscript by Mr. Dolan.

Changes Found on Reciprocated Steel Chrome Plate and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, presented from manuscript by Mr. Good.

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above the Endurance Limit—James B. Duke, presented by the author.

Effect of Fatigue on Tension-Impact Resistance—William H. Hoppmann II, presented by Mr. Noah A. Kahn.[8]

An Hypothesis for the Determination of Accumulative Damage in Fatigue—F. E. Richart, Jr., and N. M. Newmark, presented from manuscript by Mr. Richart.

ELEVENTH SESSION—SYMPOSIUM ON MINERAL AGGREGATES[3]

THURSDAY, JUNE 24, 9:30 A.M.

SESSION CHAIRMAN: K. B. WOODS

Committee D-4 on Road and Paving Materials:

Report presented by W. J. Emmons, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Asphaltic Mixtures for Sheet Asphalt Pavements

Spec. for Emulsified Asphalt

Methods for Sampling Bituminous Paving Mixtures

Accepted as Tentative, Revisions in:

Methods of Sampling Bituminous Materials (D 140-46 T), jointly with Committee D-8 on Waterproofing and Roofing Materials

---

[8] Published in ASTM BULLETIN, No. 155, December, 1948, p. 36.

Spec. for Hot-Mixed, Hot-Laid Asphaltic Concrete Base and Surface Courses (D 947-47 T)
Spec. for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544-41)
Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545-41)

Adopted as Standard:

Spec. for Calcium Chloride (D 98-46 T)
Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75-46 T)
Methods of Sampling and Testing Calcium Chloride (D 345-46 T)
Test for Sulfonation Index of Road Tars (D 872-46 T)
Test for Distillation of Cut-Back Asphaltic Products (D 402-47 T), as revised, to replace Standard D 402-36

Adopted as Standard, Revisions in:

Spec. for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544-41)
Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545-41)
Test for Amount of Material Finer than No. 200 Sieve in Aggregates (C 117-37), jointly with Committee C-9 on Concrete and Concrete Aggregates.

Withdrawal of Standard:

Test for Distillation of Cut-Back Asphaltic Products (D 402-36)

Symposium on Mineral Aggregates:[3]

Introduction by K. B. Woods.
Distribution of Mineral Aggregates—K. B. Woods, presented from manuscript by the author.
Petrographic and Mineralogic Characteristics of Aggregates—Roger Rhoades and Richard C. Mielenz, presented from manuscript by Mr. Douglas McHenry.
Physical and Chemical Tests and Their Significance—Harold S. Sweet, presented from manuscript by the author.
Sampling of Mineral Aggregates—C. E. Proudley, presented from manuscript by the author.
Production and Manufacture of Fine and Coarse Aggregates—Nathan C. Rockwood, presented from manuscript by the author.
Grading of Aggregates for Bituminous Construction—Jewell R. Benson, presented from manuscript by Mr. W. H. Price.
Grading of Mineral Aggregates for Portland Cement, Concrete, and Mortars—Walter H. Price, presented from manuscript by the author.
Influence of Mineral Aggregates on the Strength and Durability of Concrete—C. W. Allen, presented from manuscript by the author.
Lightweight Aggregates—R. E. Davis and J. W. Kelly, presented from manuscript by Mr. Kelly.
Mineral Aggregates for Bituminous Construction—J. T. Pauls and C. A. Carpenter, presented by Mr. Carpenter.
Mineral Aggregates for Low-Cost Roads and Water-Bound Macadams—Edward A. Willis and James A. Kelly, Jr., presented by Mr. Willis.
Mineral Aggregates for Railroad Ballast—A. T. Goldbeck, presented by the author.
Mineral Aggregates in the Chemical and Processing Industries and in Certain Other Uses—Herbert F. Kriege, presented by the author.
Needed Research—D. O. Woolf, presented from manuscript by the author.

TWELFTH SESSION—PANEL DISCUSSION ON CORROSION OF PRESSURE VESSELS

THURSDAY, JUNE 24, 9:30 A.M.

SESSION CHAIRMAN: L. DREW BETZ

Committee D-19 on Industrial Water:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Test for Nitrate Ion in Industrial Waters
Test for Sulfate-Reducing Bacteria in Industrial Waters

Accepted as Tentative, Revisions in:

Rec. Practice for Field Sampling of Water-Formed Deposits (D 887-46 T)
Test for Total Aluminum and Aluminum Ion in Industrial Waters (D 857-45 T)
Method of Field Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (D 807-46 T)

Rec. Practice for Corrosion Tests in Industrial Water (NDHA Method) (D 935 - 47 T)

Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 - 43)

Rec. Practice for Sampling Boiler Water from Stationary Boilers (D 860 - 47)

Joint Research Committee on Boiler Feedwater Studies:

Oral progress report presented by S. F. Whirl, first vice-chairman.

Panel Discussion on the Influence of Non-Ferrous Metals and Their Compounds on the Corrosion of Pressure Vessels

Station Design and Composition of Materials as Factors in Boiler Corrosion—R. B. Donworth, presented by the author.

Corrosion of High-Pressure Steam Generators: Status of Our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, presented from manuscript by the author.

THIRTEENTH SESSION—METHODS OF TESTING

THURSDAY, JUNE 24, 2 P.M.

SESSION CHAIRMAN: P. G. McVETTY

Committee E-1 on Methods of Testing:

Report presented from manuscript by J. R. Townsend, chairman.

Committee D-13 on Textile Materials:

Report presented, in the absence of the chairman, by M. D. Huber, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Planning Interlaboratory Testing of Textile Materials

Accepted as Tentative, Revisions in:

Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 47 T)

Spec. for Bleached Wide Cotton Sheeting (D 503 - 40 T)

Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 - 40 T)

Spec. for Mechanical Roll Felt (D 944 - 47 T)

Def. of Terms Relating to Textile Materials (D 123 - 47 T)

Spec. for 0.007-in. Cotton Tape for Electrical Purposes (D 335 - 36)

Method of Tests and Tolerances for Woolen Yarns (D 403 - 44)

Method of Tests and Tolerances for Worsted Yarns (D 404 - 44)

Spec. and Methods of Tests for Asbestos Yarns (D 299 - 42)

Methods of Testing Felt (D 461 - 47)

Test for Fiber Length of Wool (D 519 - 40)

Adopted as Standard:

Def. of Terms Relating to Textile Materials (D 123 - 47 T), definitions of terms "azlon," "duck," and "fabric, woven narrow"

Methods of Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes (D 681 - 42 T), as revised.

Adopted as Standard, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 - 47), deletion of definition of term "glass" and addition of a note

Editorial Changes Accepted in:

Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 47 T)

Tests for Rayon Staple (D 540 - 44)

Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 - 44)

Test for Strength of Rayon Woven Fabric When Wet (D 415 - 38)

Test for Maximum Residual Shrinkage of Silk and Rayon Woven Fabrics (D 416 - 39)

Test for Colorfastness of Dyed or Printed Wool, Silk, or Rayon Fabrics to Laundering or Domestic Washing (D 436 - 37)

Test for Colorfastness of Dyed Cellulose Acetate Rayon to Atmospheric Fumes (D 682 - 47 T)

Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 42)

Methods of Quantitative Analysis of Textiles (D 629 - 46 T)

Committee E-8 on Nomenclature and Definitions:

Report presented from manuscript by P. V. Faragher, chairman.

Committee E-2 on Spectrographic Analysis:

Report presented from manuscript, in the absence of the chairman, by M. D. Huber.

Paper:

A Method of Calibrating Extensometers—W. C. Aber and F. M. Howell, presented from manuscript by Mr. Howell.[9]

Symposium on Speed of Testing:

Testing Speed Limitations for Committee A-1 Specifications for Steel—Lawford H. Fry, presented by Mr. L. H. Winkler.

The Effect of Speed of Testing on Magnesium-Base Alloys—A. A. Moore, presented from manuscript by the author.

Speed of Testing Wood: Factors in Its Control and Its Effect on Strength—L. J. Markwardt and J. A. Liska, presented by Mr. Liska.

Effect of Speed of Test Upon Strength Properties of Plastics—Albert G. H. Dietz, W. J. Gailus, and S. Yurenka, presented from manuscript by Mr. Dietz.

Methods and Equipment for Controlling Speed of Testing—Lawrence K. Hyde, presented from manuscript by the author.

FOURTEENTH SESSION—SYMPOSIUM ON METHODS AND PROCEDURES USED IN IDENTIFYING REACTIVE MATERIALS IN CONCRETE

THURSDAY, JUNE 24, 3:30 P.M.

SESSION CHAIRMAN: R. E. DAVIS

Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, presented by title.

A Wetting and Drying Test for Predicting Cement-Aggregate Reaction—C. H. Scholer, presented by title. [Withheld from publication in 1948.–ED.]

Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, presented by Mr. H. E. Davis.

Tests Used by Bureau of Reclamation for Identifying Reactive Concrete Aggregates—R. C. Mielenz and L. P. Witte, presented from manuscript by Mr. H. S. Meissner.

A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and T. R. Smith, presented from manuscript by Mr. Woolf.

Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, presented from manuscript by the author.

FIFTEENTH SESSION—MARBURG LECTURE; DUDLEY MEDAL AWARD RICHARD L. TEMPLIN AWARD

THURSDAY, JUNE 24, 8:00 P.M.

RACKHAM MEMORIAL BUILDING

SESSION CHAIRMAN: PRESIDENT T. A. BOYD

In introducing the Twenty-second Marburg Lecturer, President Boyd recalled that the purpose of the lecture was to have described at annual meetings of the Society, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of one of the functions of the A.S.T.M. in the promotion of knowledge of materials, the lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society, who placed its work on a firm foundation and through his development of the technical programs brought wide recognition to the A.S.T.M. as a forum for the discussion of properties and tests of engineering materials.

President Boyd then presented Dr. P. C. Aebersold, Chief of the Isotopes Division, Atomic Energy Commission, Oak Ridge, Tenn., who delivered the lecture on "Isotopes and Their Application in the Field of Industrial Materials."[10] The lecture pointed out among other things, how tracer atoms may be used in research aimed at improvement of material and increased efficiency of production. They are already being used widely in investigations covering such varied topics as friction, oil well logging, ventilation, steelmaking, vulcanization, polymerization, welding, corrosion, catalysis, diffusion, chemical exchange, and pharmaceutical syntheses.

---

[9] Published in ASTM BULLETIN, No. 155, December, 1948, p. 33.

[10] See p. 527; also available as a separate publication.

President Boyd, in expressing appreciation to Dr. Aebersold for his very timely and instructive lecture on a subject of particular interest to the A.S.T.M. presented to him on behalf of the Society, The Edgar Marburg Lecture Certificate.

Award of Charles B. Dudley Medal:

The chairman recognized H. J. Gilkey, chairman of the Committee on Award of the Charles B. Dudley Medal. Mr. Gilkey presented as the recipients of the Twentieth Award of the Dudley Medal, Messrs. P. R. Toolin, Research Engineer, Mechanics Department, Westinghouse Research Laboratories and N. L. Mochel, Manager, Metallurgical Department, Westinghouse Electric Corp. for their paper on "The High - Temperature Fatigue Strength of Several Gas Turbine Alloys" presented at the 1947 Annual Meeting and published in the 1947 *Proceedings*. Mr. Gilkey stated that this annual award of the Dudley Medal is made to the author of a technical paper of outstanding merit which is considered as constituting an original contribution on research in engineering materials. The Award commemorates the name of the first president of the Society, Charles B. Dudley, and it is also intended as a means of stimulating research in materials and of recognizing meritorious contributions to the Society. On behalf of and as the representative of the Society, President Boyd then made the Twentieth Award of the Charles B. Dudley Medal to Messrs. Toolin and Mochel.

Mr. Toolin, in receiving this award, expressed sincere appreciation of the honor conferred upon himself and Mr. Mochel.

Richard L. Templin Award:

The chairman recognized L. W. Teller, chairman of the Committee on the Richard L. Templin Award. Mr. Teller presented Mr. A. G. H. Dietz, representing himself and Messrs. G. S. Burr, W. J. Gailus, J. O. Silvey, and S. Yurenka, Massachusetts Institute of Technology, to whom the third Richard L. Templin Award was made for their paper on "Universal Plastics Testing Machine" published in the 1947 *Proceedings* and in the December, 1947, issue of the ASTM BULLETIN. Mr. Teller stated that the purpose of the award is to stimulate research in the development of testing methods and apparatus, to encourage the presentation to the Society of papers describing new and useful testing procedures and apparatus, and to recognize meritorious efforts of this character. The award was established in 1945 when the Executive Committee received a gift from Richard L. Templin to be used for a prize award for papers describing new testing methods and apparatus. Vice-President Richard L. Templin then made the third Richard L. Templin award.

In accepting this award, Mr. Dietz expressed the appreciation of the authors for this honor conferred upon them.

SIXTEENTH SESSION—FUELS, FIRE TESTS, BUILDING CONSTRUCTIONS, ETC.

FRIDAY, JUNE 25, 9:30 A.M.

SESSION CHAIRMAN: ALBERT G. H. DIETZ

Committee D-3 on Gaseous Fules:

Report presented, in the absence of the chairman, by W. R. Fraser, and the following action taken:

Adopted as Standard:

Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 - 46 T), as revised.

Committee D-5 on Coal and Coke:

Report presented by A. C. Fieldner, chairman, and the following actions taken:

Accepted as Tentative:

Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances

Accepted as Tentative, Revisions in:

Method of Drop Shatter Test for Coal (D 440 - 37 T)

Adopted as Standard, Revisions in:

Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 46)
Methods of Sampling Coals Classed According to Ash Content (D 492 - 46)
Drop Shatter Test for Coke (D 141 - 23)

Committee D-6 on Paper and Paper Products:

Report presented by G. H. Harnden, secretary, and the following actions taken:

Accepted as Tentative:

Test for Reducible Sulfur in Paper
Test for Analytical Filter Papers
Test for Stretch of Paper and Paper Products Under Tension
Test for Organic Nitrogen in Paper and Paperboard,
Test for Measuring Paraffin Wax Absorptiveness of Paper
Test for 45 Degree, 0 Degree Directional Reflectance for Blue Light (Brightness) of Paper
Test for Water-Vapor Permeability of Paper, Paperboard, and Other Sheet Materials, which represents a consolidation and replacement of Tentative Methods D 783 and D 830
Method of Preparation of Magnesium-Oxide Standard for Spectral Reflectivity

Adopted as Standard:

Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 - 46 T)
Test for Resistance of Paper to Passage of Air (D 726 - 46 T)
Test for Tensile Breaking Strength of Paper and Paper Products (D 828 - 45 T)
Test for Wet Tensile Breaking Strength of Paper and Paper Products (D 829 - 45 T)
Test for Ply Adhesion of Paper or Vulcanized Fibre (D 825 - 46 T), as revised.

Withdrawal of Tentatives:

Test for Water Vapor Permeability of Paper and Paperboard (D 783 - 44 T)
Test for Water Vapor Permeability of Paper and Other Sheet Materials at Elevated Temperature and Humidity (D 830 - 45 T)

Editorial Change in Standard:

Test for Thickness of Paper and Paper Products (D 645 - 43), changing the scope of Method A as stated in Section 1 to read as follows by the addition of the italicized words: "Method A, for the general run of papers, paper products, and *for container grades of paperboard.*"

Accepted for Publication as Information:

Proposed Method of Wax Pick Test for Surface Strength of Paper

Committee D-7 on Wood:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following action taken:

Adopted as Standard:

Methods of Testing Small Clear Specimens of Timber (D 143 - 47 T)

Committee D-10 on Shipping Containers:

Report presented from manuscript, in the absence of the chairman, by L. C. Gilbert.

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Accepted as Tentative:

Method for Sampling and Chemical Analysis of Borax

Adopted as Standard:

Def. of Terms Relating to Soaps and Other Detergents (D 459 – 47 T)

Adopted as Standard, Revisions in:

Spec. for Compound Chip Soap (with Rosin) (D 690 – 44)
Spec. for Milled Toilet Soap (D 455 – 39)
Spec. for White Floating Toilet Soap (D 499 – 39)

Reaffirmation of Standards:

Spec. for Chip Soap (D 496 – 39)
Spec. for Ordinary Bar Soap (D 497 – 39)
Spec. for Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39)
Spec. for Alkaline Soap Powder (D 534 – 42)
Spec. for Palm Oil Solid Soap (Type A, Straight; Type B, Blended) (D 535 – 41)
Spec. for Palm Oil Chip Soap (Type A, Straight; Type B, Blended) (D 536 – 42)
Spec. for Sodium Metasilicate (D 537 – 41)
Spec. for Olive Oil Solid Soap (Type A, Straight; Type B, Blended) (D 592 – 42)
Spec. for Salt-Water Soap (D 593 – 42)
Spec. for Sodium Sesquisilicate (D 594 – 41)
Spec. for Olive Oil Chip Soap (Type A, Straight; Type B, Blended) (D 630 – 42)
Test for Particle Size of Soaps and Other Detergents (D 502 – 39)

Editorial Changes Accepted in:

Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 46)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Accepted as Tentative, Revisions in:

Fire Tests of Building Construction and Materials (E 119 – 47)
Test for Combustible Properties of Treated Wood by the Crib Test Method (E 160 – 46)
Test for Combustible Properties of Treated Wood by the Fire-Tube Test Method (E 69 – 47)

Committee E-6 on Methods of Testing Building Constructions:

Report presented, in the absence of the chairman, by L. C. Gilbert.

Paper:

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method—J. Jonakin, P. Cohen, R. Corey, and B. Jain, presented from manuscript by Mr. Jonakin.

SEVENTEENTH SESSION—CREEP AND FATIGUE—NON-FERROUS

FRIDAY, JUNE 25, 9:30 A.M.

DETROIT-LELAND HOTEL

SESSION CHAIRMAN: VICE-PRESIDENT R. L. TEMPLIN

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by A. W. Tracey, secretary. Mr. Tracey called upon L. J. Gorman to present the report of Subcommittee VIII on Galvani and Electrolytic Corrosion.

Appendix

Apparatus and Factors in Salt Fog Testing—V. M. Darsey and W. R. Cavanagh, presented by Mr. Darsey.

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented from manuscript by R. B. Saltonstall, secretary.

Committee B-1 on Wires for Electrical Conductors:

Report presented by J. H. Foote, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Rolled Aluminum Rods (EC Grade) for Electrical Purposes
Spec. for Cored, Annular, Concentric-Lay Stranded Copper Conductors
Spec. for Hard-Drawn Copper-Covered Steel Wire
Spec. for Concentric-Lay Stranded Copper-Covered Steel Conductors
Spec. for Concentric-Lay Stranded Copper and Copper Covered Steel Composite Conductors
Spec. for Hard-Drawn Aluminum Wire for Electrical Purposes

Spec. for Concentric-Lay Stranded Aluminum Conductors, Hard-Drawn
Spec. for Concentric-Lay Stranded Aluminum Conductors, Steel-Reinforced (ACSR)

Accepted as Tentative, Revisions in:

Test for Resistivity of Copper and Copper Alloy Electrical Conductors (B 193 - 45 T)
Spec. for Concentric-Lay Stranded Copper Conductors, Hard, Medium-Hard or Soft (B 8 - 46)
Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 - 45)

Adopted as Standard:

Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 45 T)

Editorial Changes Accepted in:

Spec. for Hard-Drawn Copper Wire (B 1 - 47)
Spec. for Medium Hard-Drawn Copper Wire (B 2 - 47)
Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 - 46)
Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Purposes (B 48 - 45)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 45 T)
Spec. for Bronze Trolley Wire (B 9 - 46)
Spec. for Copper Trolley Wire (B 47 - 46)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by E. E. Thum, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for Slab Zinc (Spelter) (B 6 - 46)

After presentation of the report by Mr. Thum, the meeting adopted a resolution recognizing the outstanding service that he had rendered to Committee B-2, and to the Society, in the 20 years in which he had served as secretary of the committee from 1928 to 1939 and since then as chairman.

Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

Report presented, in the absence of the chairman, by W. A. Gatward, and the following actions taken:

Accepted as Tentative:

Test for Modulus of Elasticity of Thermostat Metals (Cantilever Method)

Accepted as Tentative, Revisions in:

Test for Resistivity of Metallic Materials (B 63 - 36), including certain changes in dimensions of specimen shown in Fig. 2.
Method of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 - 42), including a correction in the Note in Fig. 1, changing "0.0254 in." to read "0.0250 in."

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by G. H. Harnden, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Naval Brass Rods, Bars, and Shapes (B 21 - 47 T)
Spec. for Brass Plate, Sheet and Strip (B 36 - 47 T)
Spec. for Seamless Copper Tubes (B 75 - 47 T)
Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet and Strip (B 122 - 47 T)
Spec. for Copper Rods, Bars, and Shapes (B 133 - 47 T)
Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 47 T)
Spec. for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 47 T)
Spec. for Copper-Nickel-Zinc Alloy Rods and Bars (B 151 - 47 T)
Spec. for Copper Sheet, Strip, and Plate (B 152 - 47 T)
Spec. for Copper Bus Bars, Rods, and Shapes (B 187 - 47 T)
Spec. for Beryllium-Copper Alloy Rod and Bar (B 196 - 47 T)
Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes (B 220 - 47 T)
Spec. for Copper Rods for Locomotive Staybolts (B 12 - 47)
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 47)
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 47)
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 47)
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 47)

Adopted as Standard:

Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 - 46 T)
Spec. for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 - 46 T)
Spec. for Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 46 T)

Spec. for Aluminum Bronze Sand Castings (B 148 - 46 T)
Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 - 46 T)
Spec. for Silicon-Bronze and Silicon-Brass Sand Castings (B 198 - 46 T)
Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 45 T), as revised.
Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 46 T), as revised.
Spec. for High-Leaded Tin-Bronze Sand Castings (B 144 - 46 T), as revised.
Spec. for High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 - 46 T), as revised.

Adopted as Standard, Revisions in:

Spec. for Phosphor Copper (B 52 - 46)
Spec. for Copper Water Tube (B 88 - 47)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 47)
Spec. for Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 47)
Spec. for Brass Wire (B 134 - 47)
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 47)
Spec. for Copper-Alloy Condenser Tube Plates (B 171 - 47)

Editorial Changes Accepted in:

The addition of the Note as preprinted was accepted for inclusion in the following specifications as an editorial change:

B 19 - 47 T
B 21 - 47 T
B 36 - 47 T
B 75 - 47 T
B 121 - 47 T
B 122 - 47 T
B 129 - 47 T
B 130 - 47 T
B 131 - 47 T
B 133 - 47 T
B 135 - 47 T
B 139 - 47 T
B 150 - 47 T
B 151 - 47 T
B 152 - 47 T
B 159 - 47 T
B 169 - 47 T
B 187 - 47 T
B 188 - 47 T
B 194 - 47 T
B 195 - 47 T
B 196 - 47 T
B 197 - 47 T
B 206 - 47 T
B 12 - 47
B 13 - 41
B 14 - 18
B 16 - 47
B 42 - 47
B 43 - 47
B 68 - 47
B 88 - 47
B 96 - 47
B 97 - 47
B 98 - 47
B 99 - 47
B 100 - 47
B 103 - 47
B 111 - 47
B 124 - 47
B 134 - 47
B 138 - 47
B 140 - 47
B 171 - 47

Committee B-6 on Die-Cast Metals and Alloys:

Report presented, in the absence of the chairman, by I. V. Williams, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Die Castings (B 85 - 46 T)
Spec. for Magnesium-Base Alloy Die Castings (B 94 - 47 T)
Spec. for Copper-Base (Brass) Alloy Die Castings (B 176 - 47 T)

Adopted as Standard, Revisions in:

Spec. for Zinc-Base Alloy Die Castings (B 86 - 46), except to change the second paragraph of the Appendix to read as follows: "In the tension testing of die-cast zinc-alloy specimens, the rate of straining shall not exceed 0.25 in. per in. per min. measured on the gage length of the specimen.
Spec. for Lead- and Tin-Base Alloy Die Castings (B 102 - 44)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum and Aluminum-Alloy Sheets and Plate (B 209 - 46 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubing (B 210 - 46 T)
Spec. for Aluminum and Aluminum-Alloy Bars, Rods, and Wire (B 211 - 47 T)
Spec. for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 47 T)
Spec. for Magnesium-Base Alloy Extruded Round Tubing (B 217 - 46 T)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 47 T)

Editorial Changes Accepted in:

The addition of a new section on Sampling for Chemical Analysis to be included in all of the magnesium specifications, and also the note proposed for incorporation in each specification under the jurisdiction of Committee B-7 were accepted in the form as preprinted as editorial changes.

In presenting the report, Mr. Williams called attention to a change under the activities of Subcommittee VII. The third sentence was changed from the form as preprinted, namely, "but was rejected in favor of retention of the present code systems." to read: "and 41 of the

96 individuals on the committee returned ballots. Twenty-two favored the new system and 13 favored continuation of the present system. As a result, the matter was referred back to Subcommittee VII for further consideration."

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by W. A. Reich, chairman, and the following actions taken:

Adopted as Standard:

Test for Apparent Density of Metal Powders (B 212 - 46 T)
Method of Test for Flow Rate of Metal Powders (B 213 - 46 T)
Test for Sieve Analysis of Granular Metal Powders (B 214 - 46 T)
Method of Sampling Finished Lots of Metal Powders (B 215 - 46 T)

Committee E-3 on Chemical Analysis of Metals:

Report presented by D. R. Evans, chairman-elect, and the following actions taken:

Adopted as Standard:

Chemical Analysis of Metallic Materials for Electrical Heating (E 38 - 42 T)
Chemical Analysis of Copper (Electrolytic Determination of Copper) (E 53 - 46 T)
Method of Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (E 55 - 46 T), with the following editorial revisions:

Section 4 (a).—Delete the words "with shears" at the end of the first sentence. In the second sentence, change "0.010 in." to read "0.040 in."

Section 6 (a).—Change to read: "The prepared sample shall weigh three times the amount required for the analysis and shall be divided into three equal parts, each of which shall be placed in a package and sealed, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary."

Method of Sampling Slab Zinc (Spelter) (E 65 - 46 T)
Rec. Practices for Apparatus and Reagents for Chemical Analysis of Metals (E 50 - 46 T)

Papers:

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, presented by Mr. Gohn.
Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, presented by Mr. Phelps.
Fatigue Properties of Some Coppers and Copper Alloys in Strip Form—H. L. Burghoff and A. I. Blank, presented by Mr. Blank.
Tensile Creep, and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, presented from manuscript by the author.

EIGHTEENTH SESSION—CEMENT, CONCRETE, AND CONCRETE AGGREGATES

FRIDAY, JUNE 25, 2 P.M.
DETROIT-LELAND HOTEL
SESSION CHAIRMAN: F. E. RICHART

Committee C-1 on Cement:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative:

Definition of the Term Addition

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of Portland Cement (C 114 - 46 T), comprising new methods for determining titanium oxide in portland cement.
Spec. for Air-Entraining Portland Cement (C 175 - 47 T)
Spec. for Portland Blast-Furnace Slag Cement (C 205 - 47 T)
Spec. for Portland Cement (C 150 - 47)

Adopted as Standard:

Spec. for Masonry Cement (C 91 - 44 T)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by K. B. Woods, chairman and the following actions taken:

Adopted as Standard:

Spec. for Ready-Mixed Concrete (C 94 - 47 T)
Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 - 46 T), as revised.

Adopted as Standard, Revisions in:

Test for Organic Impurities in Sands for Concrete (C 40 - 33)

Test for Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 - 37), jointly with Committee D-4 on Road and Paving Materials.

Sanford E. Thompson Award:

Mr. Richart then introduced Mr. Sanford E. Thompson who made the seventh Sanford E. Thompson Award to Mr. W. C. Hanna, Chief Chemist and Chemical Engineer, California Portland Cement Co., for his paper on "Unfavorable Chemical Reactions of Aggregates in Concrete and a Suggested Corrective" published in the 1947 *Proceedings*. This award was established in 1938 by Committee C-9 on Concrete and Concrete Aggregates as an annual token of recognition to the author of a paper of outstanding merit on concrete and concrete aggregates presented at an annual meeting of the Society. In making the award, Mr. Thompson stated that Mr. Hanna's paper was selected because of the wealth of information which it contains on the difficulties experienced with aggregates in defective concrete and for the suggested methods for correcting the effect of the reactive aggregate.

In accepting the certificate of the Sanford E. Thompson Award, Mr. Hanna expressed sincere appreciation of the honor and recognition conferred upon him and said that he was particularly gratified to receive this award personally from Mr. Thompson.

Committee C-11 on Gypsum:

Report presented by C. E. Abbey, secretary-elect, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Gypsum Plasters (C 28 - 40)

Adopted as Standard:

Def. of Terms Relating to Gypsum (C 11 - 41 T)

Withdrawal of Tentative Revision of Standard:

Methods of Testing Gypsum and Gypsum Products (C 26 - 42)

Spec. for Calcined Gypsum for Dental Plasters (C 72 - 40)

Withdrawal of Standards:

Spec. for Calcined Gypsum for Dental Plasters (C 72 - 40)

Spec. for Gypsum Pottery Plaster (C 60 - 40)

Committee C-16 on Thermal Insulating Materials:

Report presented by Ray Thomas, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Water Vapor Permeability of Sheet Materials Used in Connection with Thermal Insulation (C 214 - 47 T)

Test for Bulk Density of Thermal Insulating Cement (C 164 - 44)

Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (C 166 - 45)

Def. of Terms Relating to Thermal Insulating Materials (C 168 - 47)

Adopted as Standard:

Spec. for Structural Insulating Board Made from Vegetable Fibers (C 208 - 46 T)

Spec. for 85 per cent Magnesia Thermal Insulating Cement (C 193 - 44 T), as revised.

Spec. for Asbestos Thermal Insulating Cement (C 194 - 44 T), as revised.

Spec. for Mineral Wool Thermal Insulating Cement (C 195 - 47 T), as revised.

Spec. for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196 - 47 T), as revised.

Spec. for Diatomaceous Silica Thermal Insulating Cement (C 197 - 47 T), as revised.

Methods of Testing Structural Insulating Board Made from Vegetable Fibers (C 209 - 46 T), as revised.

Adopted as Standard, Revisions in:

Def. of Terms Relating to Thermal Insulating Materials (C 168 - 47)

Papers:

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., presented from manuscript by Mr. Hasler.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—Walter H. Price and John W. Robison, presented by Mr. Price.

Research on Concrete Durability as Affected by Coarse Aggregate—Harold S. Sweet, presented from manuscript by the author.

The Effect of Repeated Loading on the Bond Strength of Concrete—Supplement 1.—C. W. Muhlenbruch, presented from manuscript by the author.

NINETEENTH SESSION—RUBBER, PLASTICS, INSULATING MATERIALS, AND ENGINE ANTIFREEZES

FRIDAY, JUNE 25, 2 P.M.

SESSION CHAIRMAN: PRESIDENT T. A. BOYD

Committee D-15 on Engine Antifreezes:

Report presented by E. H. Keller, secretary.

Committee D-11 on Rubber and Rubber-Like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Test for Volume Resistivity of Electrically Conductive Rubber and Rubber-Like Materials

Accepted as Tentative, Revisions in:

Spec. for Friction Tape for General Use for Electrical Purposes (D 69 – 46 T)
Spec. for Rubber Insulating Tape (D 119 – 46 T)
Methods of Testing Rubber Insulated Wire and Cable (D 470 – 46 T)
Methods of Testing Automotive Air Brake and Vacuum Brake Hose (D 622 – 44 T)
Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by the Test Tube Method (D 865 – 47)

Adopted as Standard, Revisions in:

Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 41)
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 42)
Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 45)
Method of Air Pressure Heat Test of Vulcanized Rubber (D 454 – 41)
Test for Tear Resistance of Vulcanized Rubber (D 624 – 44)

Editorial Changes Accepted in:

Methods of Testing Rubber Adhesives (D 816 – 46 T)

Accepted for Publication as Information Only:

Method for Measuring the Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus

It was announced that the committee had withdrawn from the report the proposed revisions in the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 46 T). These revisions together with the new Tentative Specifications for Thermoplastic Vinyl Polymer Sheaths for Electrical Insulated Cords and Cables will be presented to the Society through the Administrative Committee on Standards subsequent to the Annual Meeting. The Proposed Specifications and Methods of Test for Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex will also be submitted to the Society for publication as information subsequent to the Annual Meeting.

Committee D-20 on Plastics:

Report presented by B. L. Lewis, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Stiffness in Flexure of Nonrigid Plastics (D 747 – 43 T)
Spec. for Laminated Thermosetting Materials (D 709 – 47 T), jointly with Committee D-9
Spec. for Methacrylate Molding Compounds (D 788 – 44 T)
Spec. for Polystyrene Molding Compounds (D 703 – 44 T)
Spec. for Cellulose Acetate Compounds (D 706 – 46 T)
Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 44 T), subject to concurrence of Committee D-9 on Electrical Insulating Materials
Rec. Practice for Molding Specimens of Phenolic Materials (D 796 – 45 T)

It was announced that the committee had withdrawn from the report as preprinted the proposed revisions in the Tentative Specifications for Nonrigid Vinyl Chloride Plastics (D 744 - 44 T) and in the Tentative Definitions of Terms Relating to Plastics (D 883 - 46 T).

Adopted as Standard:

Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (D 758 - 44 T)
Methods of Test for Tensile and Compressive Properties of Plastics at Subnormal and Supernormal Temperatures (D 759 - 44 T)
Method of Long-Time Tension Tests of Plastics (D 674 - 46 T)
Test for Tensile Strength of Molded Electrical Insulating Materials (D 651 - 42 T), jointly with Committee D-9
Test for Deviation of Line of Sight Through Transparent Plastics (D 881 - 46 T)
Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 - 44 T) and Method of Designating the Flow Temperature of Thermoplastic Molding Materials (D 863 - 45 T), revised and combined into a single standard.
Test for Estimating Blocking of Plastic Sheets (D 884 - 46 T), as revised.

Committee D-14 on Adhesives:

Report presented from manuscript, in the absence of the chairman, by P. J. Smith.

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

Adopted as Standard:

Def. of Terms Relating to Naval Stores and Related Products (D 804 - 46 T)
Methods of Sampling and Testing Dipentene (D 801 - 46 T), as revised, with a correction in the revised Section 14 (*a*), the flask should be 1000-ml. rather than 100-ml. capacity

Committee D-9 on Electrical Insulating Materials:

Report presented by R. W. Orr, chairman, and the following actions taken:

Accepted as Tentative:

Test for Detection of Free Sulfur in Electrical Insulating Oils, with the following changes:

Section 1.—Omit the reference to Note 2.

Section 2.—Add the following at the end of the definition in this section; "It will detect any free sulfur that is present, but it may also detect sulfur derived from some sulfur compounds which may react under the conditions of the test."

Section 6 (c).—Add the following after the first sentence, "Discoloration may also be due to the corrosive action of sulfur derived from some combined sulfur compounds reacting under the conditions of the test."

In the second paragraph, replace the last sentence by the following: "If no precipitate forms in 7 hr. under the test conditions, free sulfur shall be considered absent. Conversely, formation of a precipitate is not positive proof of the presence of free sulfur. It may simply indicate that some combined sulfur has reacted under the test conditions. However, formation of a precipitate shall be interpreted as failure of the oil to pass the test."

Note 2.—Change the phrase "which may cause trouble in transformers" to read, "which may cause trouble in electrical apparatus, particularly transformers."

Accepted as Tentative, Revisions in:

Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 - 42)
Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 46 T)
Spec. for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 - 47 T)
Spec. for Laminated Thermosetting Materials (D 709 - 47 T), jointly with Committee D-20
Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 45)
Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 - 44)

Adopted as Standard:

Test for Tensile Strength of Molded Electrical Insulating Materials (D 651 - 42 T), jointly with Committee D-20 on Plastics
Test for Gas Content of Insulating Oils (D 831 - 45 T)

Adopted as Standard, Revisions in:

Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 45), adoption only of revisions in Table I and Section 14(*a*).

Withdrawal of Tentatives:

Test for Compressive Strength of Electrical Insulating Materials (D 649 - 42 T)
Test for Flexural Strength of Electrical Insulating Materials (D 650 - 42 T)

It was announced that the committee had withdrawn from the report as preprinted, the proposed revisions in the Tentative Specifications for Nonrigid Vinyl Chloride Plastics (D 744 - 44 T). Also the committee withdrew the recommendation for adoption as standard of the Tentative Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin (D 877 - 46 T).

Several changes in the main text of the report under the activities of Subcommittee IV on Liquid Insulation were also accepted.

Papers:

Requirements of an Engine Anti-Freeze and Methods of Evaluation—D. H. Green, J. C. Kratzer, and P. I. Emch, presented from manuscript by Mr. Kratzer.[11]

Nontoxic Rubber for Gas Masks—S. H. Katz, presented by the author.

The Effect of Temperature on Creep of Two Laminated Plastics—W. N. Findley, C. H. Adams, and W. J. Worley, presented from manuscript by Mr. Findley.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab, presented by Mr. Clark.

There being no further business, the Fifty-first Annual Meeting was adjourned *sine die* by President T. A. Boyd.

July, 1948.

---

[11] Published in ASTM BULLETIN, No. 154, October, 1948, p. 57.

IF DOCTOR MARBURG CAME BACK TODAY

ANNUAL ADDRESS BY THE PRESIDENT

T. A. BOYD[1]

JUNE 23, 1948

JUST thirty years ago, at the 1918 Annual Meeting of our Society, word was received of the untimely death of Dr. Edgar Marburg. Dr. Marburg, whose name is commemorated in the important Edgar Marburg Lecture established in 1925, had been secretary-treasurer of the American Society for Testing Materials ever since its organization in 1902. Illness had prevented him from going to Atlantic City for the 1918 meeting; but he had previously organized, as one of the "leading features" of that meeting, a discussion of "Cooperation in Industrial Research." He had also prepared an opening announcement for the symposium.[2]

With Dr. Marburg's active interest in research and his recognition of its importance to our Society, what would he see if he could return today? This address is an attempt to answer that question in part, and by doing so to show something of the progress of research since 1918 in the nation and in our Society. The extent of that progress would amaze anyone who returned after an absence of thirty years, and it is hoped that an account of it may be of interest to those who hear this address. It just so happens that the period spoken of coincides exactly with the era of my own activity in the field of research. So, to some degree, I am able to answer the question proposed from personal observation.

Thirty years is long enough to be the standard measure of a generation. In Mark Twain's language it is even longer, for on one occasion he said, "By forever, I mean thirty years." Needless to say, the period has seemed to me much shorter than that.

One of the first things that would strike Dr. Marburg, if he came back today, is how greatly the utilization of research by industry has been extended during the past thirty years. Back in 1918, research by industrial concerns was only in its infancy, although even then about 300 companies are said to have had research laboratories of some kind. But, of the more than 2500 industrial research laboratories in existence today, most have been organized since the First World War. Also, many of the laboratories which did exist in 1918 have grown greatly in size since that time. The combined effect of these two developments has been that the number of workers in industrial research laboratories has increased from only about 7000 in 1920 to 135,000 or more today, which is a growth of nearly twenty-fold.[3, 4]

---

[1] Research Consultant, Research Laboratories Division, General Motors Corp., Detroit, Mich.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part II, p. 5 (1918).

And so Dr. Marburg would see a great many research laboratories in the land, some large, some even huge, and some not so large. He would see that some of them are new and, to him, impressively fine, and that some are not so fine. However, he would be more interested in the men working in the laboratories than in the laboratories themselves. He would, therefore, be glad to find in industrial research today some present-day Edisons, some Marconis, some Bells, some Pasteurs, and some Baekelands.

He would see, too, that, in addition to all the research laboratories established by industry, there has been since his day a considerable increase in consulting research laboratories and in research institutes conducting investigations for industry. He would see also that today nearly 300 universities and colleges are offering research service to industry.[4]

The highest rate of growth in number of industrial research laboratories occurred in the dozen years immediately following 1918, and by 1930 there were over 1600 industrial research laboratories in the country,[5] more than half as many as today. The rapid growth during that period was no doubt due in large measure to the impulse given research by its intensive and widespread application during the years of the First World War, and to the increased realization of the importance of research which grew out of it. Now again, after the recent World War, a similar upsurge in the utilization of research by industry is under way, and it is going forward in spite of shortages of qualified personnel and of building costs that are abnormally high.

Dr. Marburg would find also, if he came back today, that the acceptance of research and the utilization of the research technique has been extended beyond the field of technology in which it developed. In the business world, for instance, research is being done on personnel and industrial relations; on production and production methods; on the market, the consumer, and marketing procedure; on economic conditions and tendencies; and on other aspects of business.[6] "It is just as important," said Alfred P. Sloan, Jr., "to apply research to all the functional activities of business as it is to the technological phases of the enterprise." Fact is that in modern business the executive who decides questions merely by the old system of snap judgment cannot long survive—or else it is the business that does not survive.

Dr. Marburg would see that, during the thirty years since his time, research has brought into being many, many new products, and that these have had a big effect upon the life of the nation. He would find a radio in every home, bringing even into the most remote places music, drama, world events, and round-table discussions—as well as singing commercials, soap operas, and the controversial blood-and-thunder

---

[3] Research—a National Resource: a Report of the National Research Council to the National Resources Planning Board, Vol. 2, Sec. 4, p. 173 (1941).

[4] "Industrial Research Laboratories of the United States," *Bulletin*, National Research Council, No. 113 Eighth Edition (1946).

[5] "Industrial Research Laboratories of the United States", *Bulletin*, National Research Council, No. 81 (1931).

[6] Research—A National Resource, III, *Business Research*, National Resources Planning Board (1941).

thriller. He would see television nowadays too, bringing visual broadcasts into many homes. He would find that since his time the motion picture has learned to talk, and that many films are dressed in color as well. He would see the number of automobiles increased since 1918 by five-fold and hard-surface highways spreading like a net over all the nation. He would see that railroad trains are air-conditioned now and therefore clean and comfortable, and that many of them are powered by diesel engines. In the field of power generation he would hear much talk nowadays about jets and turbines, and about atomic energy also. He would find that aviation has become a big industry with airports and airlines covering the nation and the world. He would see in wide use synthetic rubber and synthetic textiles of several kinds, so that, for one thing, the cotton stocking has been almost completely displaced by nylon hose. In the field of medicine he would find insulin, sulfa drugs, penicillin, streptomycin, and other marvelous life-saving remedies. These are but a few of the many changes within the thirty years just past.

The multitude of new things resulting from research, and all the modifications and improvements made in old things meanwhile by the same means, have greatly changed life in America since 1918—almost revolutionized it, in fact. As an ex-farm boy, I myself am particularly aware of the extent to which life on the farm has been changed through advances in technology. Gone from farm life are the isolation, the inconveniences, the discomforts, and much of the drudgery so universal in my years on the farm.

After the Battle of Britain, Winston Churchill uttered that historic saying of his about the handful of heroic airmen who had won it. "Never in the field of human conflict was so much owed by so many to so few," he said. So also, in view of the small number of those who are and have been engaged in research and of the large magnitude of the changes and benefits which have come out of their efforts, it seems appropriate to say after the same fashion that no other such small number of men in any field of human endeavor has influenced our way of life to a greater degree. It might even be said of the modern research laboratory, as Sir Francis Bacon wrote in *The New Atlantis* about the organization for finding new things, which he imagined there and called Solomon's House, that it is "the noblest foundation... that ever was upon the earth."

Dr. Marburg would find though, that in spite of all this, the acceptance of research by men in industry and the understanding and appreciation of its potentialities is not by any means universal, even yet. Not more than ten per cent of business enterprises have research laboratories today. There is still a widespread belief that research is so costly and so complicated that only large organizations can afford to engage in it. That belief prevails in spite of the circumstance that, although research *is* costly in time and money, it pays its own way in the long run. If that were not so, the big company could not afford to do research either.

Experience from the first has shown that industrial research, when conducted with imagination and judgment, is in fact one of the most profitable of endeavors. Thus, Charles L. Reese of the du Pont Company, in his contribution to that symposium on "Cooperation in Industrial Research" held at the Annual Meeting of our Society back in 1918, said that an expenditure of $1,200,000 on research by his company during the period from 1912 to 1915 had resulted in savings of $14,000,000.[7] That is a return of more than ten-fold. More recently, R. P. Russell, who at the time was president of the Standard Oil Development Co., said that for each one thousand dollars spent by that organization on research and development more than fifteen thousand dollars had been added to the profits of the stockholders.[8] These are just two instances among the many that might be cited.

The head of a concern who, because his business is not a large one, believes that he cannot afford to do research is thus not thinking straight. It is not necessary to maintain a large staff in order to do research. Not many of the research laboratories which existed thirty years ago had large staffs. Most were quite small then, but some have grown to be much larger since. And the reason they grew was because of demonstrated service to the business. A research laboratory should, in fact, always be started on a modest scale. The initial staff may be only one man. Nowadays the efforts of a small staff may readily be supplemented when desired by work in research institutes, in consulting research laboratories, and in research foundations conducted by educational institutions. In respect to the widely held but mistaken belief that small businesses cannot engage in research, C. E. K. Mees has said that in his opinion the small concern can do research with particular profit and advantage.[9] For one thing, as Dr. Mees suggests, the research director of such a company can be an active part of the management of the concern to a far greater extent than is possible in a big organization.

But the profit in industrial research is not limited to the company that does it, of course. Such research is profitable, too, for the user of the product, for the customer—and usually the customer is the one who benefits most of all. This is true in respect to the automobile, for instance, which over the years has been marvelously improved in comfort and utility, and the cost of it reduced at the same time. It is true of the electric light bulb, the efficiency of which has been multiplied by five and the cost divided by almost the same figure. It is true of radio, which, all within a few years, went from the crystal set with headphones to FM with its fine reception and freedom from static. It is true of most products of research, in fact.

Competitors, too, have sometimes benefited from research conducted by other members of their industry—although, to be sure, that is quite contrary to what is generally supposed. One instance of this is the research which yielded Freon, the nontoxic and noninflammable refrigerant containing fluorine. That research was done on behalf of Frigidaire, but it greatly benefited as well every maker of air-conditioning equipment—not to mention the people whom he serves.

What with technology advancing so rapidly as it is nowadays, the question of whether a company is doing research or not has become one of the most important items for the investor to look into when scrutinizing a company as a possible place to put some of his funds. Is the company doing any research at all? And, if it is, what is the character of that research? Is it merely the fixing or "bug-hunting" type of investigation? Or is some of the company's experimentation directed toward finding out what it is going to do when it can't do just what it is doing now? In evaluating the probable soundness of an investment in industry, the answer to these questions can be far more important than the figures on balance sheets. For today the situation in which business finds itself sometimes is like that strange country of which, in "Through the Looking Glass," the Queen said to Alice that "Here, you see, it takes all the running you can do to keep in the same place."

---

[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 18, Part II, p. 32 (1918).

[8] *Chemical and Engineering News*, February 24, 1947, p. 576.

[9] *Chemical and Engineering News*, November 24, 1947, p. 3544.

As he looked around today, Dr. Marburg would find, too, that research has been, and still is, under fire from certain quarters—from some of those who consider themselves socially minded. Thus, the Bishop of Ripon proposed that science take a ten-year holiday to permit social evolution to catch up with the more rapid progress of science.[10] During the depression years of the 1930's the most common criticism of science and technology was that it had thrown people out of work in factories. The unemployment, which was so serious in the early 1930's, was produced by too many inventions, it was said, chiefly through the displacement of workers in factories by labor-saving machines. It is true, of course, that many factory men were out of work during the depression years; but only a fraction of those unemployed then were factory workers. For only about 20 per cent of all those working are employed in the manufacturing and mechanical industries. Furthermore, in spite of the large changes in manufacturing methods that have been made meanwhile, that percentage has not changed very much in more than 75 years.[11] Throughout that whole period those employed in factories have represented just about 20 per cent of the nation's workers.

However, the percentage of those working on farms has been going down steadily during that same period from more than 50 per cent of all workers to only about 20.[11] Thus it has not been in factories at all that the big reduction in workers has occurred. It has been on the farm. It's an old, old problem, though—this one of how to keep the boys down on the farm. Horace, that old Roman author of odes and other Latin literature, who wrote just 2000 years ago, complained in one of his writings that *he* could not keep help on his farm either. But powered farm machinery and modern conveniences, along with the radio, the automobile, and in some cases even the airplane also, are helping to solve that problem.

Within the past thirty years industrial research has grown to be an industry of such size that the direction or management of research laboratories has become a profession of importance. With more than 2500 industrial research laboratories in existence, and many research institutes and foundations also, the profession of research director has become a pretty large one. It is in recognition of this development that there has been formed since 1918 the Industrial Research Institute, a national society through which the members exchange information and ideas on the organization, management, and direction of industrial research.

---

[10] Robert A. Millikan, "Science and the New Civilization," Charles Scribner's Sons, New York, N. Y., p. 7 (1931).

[11] Food Information Series, U. S. Dept. of Agriculture, No. 82, February 16, 1944.

Over the thirty years just past, there has naturally been growth in the knowledge of how to organize and conduct research in industry. One of the things found to be of particular importance is that of picking the problems to work on. Another is that the success of industrial research depends to an important degree upon how much interest the management of the business takes in it. So it has been found important for the research director to have close contact with management—or, better still, for him to be a direct part of management. There has been growth in the understanding of how to direct research, or rather of the limitation that the course of successful research cannot be intimately directed from above, but that the function of a director of research is more that of providing proper personnel and facilities, along with such guidance and encouragement as he can give. There has been expansion also in the knowledge of how to do research: that there is no one best way, but that getting started to experiment and keeping on experimenting intelligently, in spite of failures and discouragements, is one thing that always produces results. It has come to be recognized that research is pretty largely a young man's game, and that the research laboratory that does not somehow keep on its staff a proper proportion of young men will pay for that failure in reduced productivity.

Out of experience has come, also, a better understanding of how to finance research in industry. Because ideas do not come by the year, it is not possible to keep books on forward-looking research in the same way as in other and more predictable lines of endeavor. There has been an increase in the understanding of how difficult it is to sell the output of research and of how time-consuming and costly it is to introduce new products; that in this phase a sample is always important; and that a new product cannot be introduced successfully without the collaboration of the men who developed it, for without them the product gets either more complicated and costly or less efficient. Also, the literature of research, far from adequate thirty years ago, has since grown to be very extensive. These are but a few of the many advances in the know-how of the business of industrial research.

Nothing said above is meant to imply, of course, that there was not, even thirty years ago, an important body of knowledge about how to conduct research. That any such implication would be false is illustrated, for one thing, by the following incident. It was in that symposium on "Cooperation in Industrial Research," held at the Annual Meeting in 1918, that the late Dr. Arthur D. Little uttered his famous epigram: "There is danger in an organization chart—danger that it may be mistaken for an organization."[12]

With the extension of research in

---

[12] *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part II, p. 22 (1918).

industry, it is no longer as true as it used to be that fundamental discoveries are always made by the pure science investigator, working usually in a university or similar institution. It would scarcely be possible to do as much research as is now being done by laboratories in industry without making some discoveries of a fundamental nature. And in recent years several such discoveries have come out of industrial research laboratories. A few of these, mentioned for the purpose of illustration, are the following: that the electron is a wave as well as a particle, an observation which became a cornerstone of modern physics; that refrigerating gases which are both nontoxic and noninflammable may be made if fluorine is one of the components; that a synthetic resin may act as a chemical exchange medium for softening water and for other uses; that the migration of tungsten from incandescent filaments is greatly retarded by the absence of water vapor; that the light transmission of lenses may be considerably improved by coating the surfaces with materials other than glass; that clouds may be precipitated by crystal seeding; that synthetic fibers may be greatly strengthened by orientation of the molecules through stretching in the course of manufacture, and that the brittleness so produced may be eliminated by proper treatment afterward. Other examples—just to raise the number of illustrations to ten— are ductile tungsten, chemical antiknock agents, and extreme-pressure lubricants.

In spite of all the many new discoveries made since Dr. Marburg's time, though, he would see that the unknown is still far, far greater than the known. He would see that as yet we have dug only a few small nuggets of knowledge out of the great mine which is waiting to be worked. And thus the future of the business of finding new things and of improving upon old things is still practically unlimited.

Of particular interest to Dr. Marburg would be the enlarged part which research has played and is playing in the affairs of the American Society for Testing Materials. Since one of the principal functions of the Society is "The promotion of knowledge of the materials of engineering," research has always played a part in its activities. As research activity in industry has been expanded, its utilization in the cooperative endeavors of the Society has increased to a like degree. For, of course, most of the research which advances A.S.T.M. projects is done in the laboratories of cooperating members. This is the tested method by which the committee work of the Society is done for the most part.

And so, as the activity of the Society through its technical committees has grown, and as the utilization of research by the participating companies has increased, the place of research in the cooperative work done through the Society has mounted until today it is very large indeed. Every specification or method of test in the A.S.T.M. Standards is based on a great deal of research, done mostly by laboratories of committee members who cooperated in preparing it. The most recent "Review of A.S.T.M. Research Activities" fills twelve pages of the ASTM BULLETIN.[13]

The place of the Society itself in

---

[13] ASTM BULLETIN, No. 146, May, 1947, p. 30.

the promotion of knowledge of the materials of engineering is principally in its extensive activity in providing facilities for holding meetings, and for the presentation and publication of the results of research and of the work of technical committees. At an Annual Meeting of the Society nowadays there will be scheduled between 300 and 400 meetings. Some of these are technical sessions and symposiums at which the results of research are presented in the form of papers. The publication program of the Society is therefore a very large one. In addition to the Book of Standards, it includes the annual Proceedings, and the ASTM BULLETIN, together with many separate compilations, books, and pamphlets. The meetings and publications, which are so extensive and which serve so well to keep members of the Society and its technical committees abreast of progress within the field of materials, are thus major functions of the Society, and therefore activities of the highest importance.

The Society has an Administrative Committee on Research, whose function broadly is to encourage and stimulate research activity within the Society. In trying to fulfill its function, the Society finds it needful sometimes to sponsor and to finance research on its own account. This is done, of course, only when it seems necessary to supplement in some way research being done by the organizations cooperating through it. To finance such programs, special funds have sometimes been contributed by industry. Instances of this are the work on corrosion, and that on fatigue and on the effect of temperature on metals.

With the purpose of further fulfilling its function by pursuing research on subjects of importance in the field of materials, but which are not of such immediate concern to specific industries, the Society set up many years ago a permanent Research Fund. And that is something about which I should like to say a word in closing this address, in the hope that it may fall on some receptive ears. For Dr. Marburg, if he came back today, would be disappointed in the size of the A.S.T.M. Research Fund. It amounts to only about forty-five thousand dollars. The income from the fund is thus about a hundred dollars a month; and, naturally, no more than minor searches can be financed with such a small sum.

It seems desirable, therefore, as was emphasized by Past-President Carpenter in his address at the Annual Meeting a year ago, that the principal of the A.S.T.M. Research Fund be augmented in every way possible. And, except for the addition of the entrance fees from new members, some of which are being put into it, the Fund is not now being enlarged at all. Among the ways in which the A.S.T.M. Research Fund might be increased is through contributions to it by members of the Society who may wish to help improve the position of the Society in respect to its ability to sponsor needed research.

Some members in the past have made personal contributions to the A.S.T.M. Research Fund. Among these was the late F. O. Clements, a Past-President and Honorary Member of the Society. Dr. Clements, whose passing occurred during the preparation of this address, not only made a liberal contribution to the Research Fund on his own part; he also suggested that an effort be made to boost the principal of the fund to five hundred thousand dollars. We are so far from having reached that suggested goal even today that it is still a good objective to work toward.

Men of means, and there are many such within the Society, contribute through their wills or otherwise to educational institutions for advancing the cause of education. Our Society is an educational institution. But it is more than that. It is also one which helps to gather the stuff which education is made of. And so, to those members of the Society who may be able to make contributions for the furtherance of knowledge, the A.S.T.M. Research Fund is suggested as meriting the fullest support that can be given to it.

ANNUAL REPORT OF THE BOARD OF DIRECTORS*

This annual report of the Board of Directors to the Society is presented to the members at a time of great activity in both standardization and research in materials, in which the members and committees of the Society are playing a leading rôle. Seldom if ever has our committee work been carried on so actively as in the year closing with this annual meeting. The need for more authoritative knowledge of the properties of materials is ever growing, and particularly so with new materials and combinations of materials, as well as new instrumentation and testing, that are being developed so rapidly. As the whole field expands, so potentially does the work of the Society, and it is particularly significant that several new committees have been formed in the past two years and others are being proposed. So important does the Board believe this matter to be that it is arranging for suitable studies to be made of possible new fields of work for the Society and of the ways in which such expansion can be most effectively undertaken.

The Board in particular wishes to commend the members and the committees for the fine spirit of accomplishment that is so evident throughout the Society. The enthusiasm with which new tasks are undertaken has been especially noted in the many committee meetings that have been held this year, and the alertness of committees in keeping abreast of developments in their fields is especially commendable.

The emphatic support of the recommendations to the Society a year ago for increasing the membership dues is most gratifying to the Board, as is also the splendid response of both company and individual members in continuing their membership under the increased dues. This action has assured the Society of a more secure financial position. It will enable us better to meet the present inflationary conditions that have so greatly increased the running expenses of the Society, and ultimately to increase substantially the services to the members and committees of the Society.

In the following pages the Board has brought together a brief recital of the principal happenings and developments of the Society year and of certain of its activities in matters of administration and policy. Particular attention is called to the section on Publications, which contains the first announcement of some important decisions on publication policies. The report cannot adequately convey the significance of the meetings that have been held in all thirteen districts of the Society, nor the importance of the constant influx of new membership into the Society and its committees. Examination of the committee reports, technical papers, and other publications that are currently being issued will serve to round out a picture of splendid accomplishments of which every member of the Society can justly be proud.

TECHNICAL ACTIVITIES

This meeting marks the culmination of what is probably the most active year in the history of the Society in pursuit of our technical activities through the work of our committees. Both in

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

the fields of research and of standardization of specifications and methods of test, it will be seen from the committee reports at this meeting that the year is one of unusual accomplishment.

The Administrative Committee on Standards follows the standardization activities of the Society and its annual report to the Board (see Appendix II) notes the developments throughout the year, including the approval in the interim between annual meetings of many new specifications and methods. The report of that committee also records studies in connection with expansion of standardization activities and with the efforts begun a year ago to improve the quality of A.S.T.M. standards from both editorial and technical points of view. The importance of closer editorial supervision by the technical committees themselves has been emphasized by the Administrative Committee, with the result that some 15 committees have established editorial subcommittees during the year bringing the total of such subcommittees to 26. In some of the smaller committees it is being found possible to handle such editorial matters through the officers or in an advisory committee. The importance of this subject is borne home very forcibly as one contemplates the steadily growing volume of committee output in the form of standards.

The Administrative Committee on Research, whose annual report to the Board appears as Appendix III, has continued its review of the research activities being carried on by the committees of the Society. The series of conferences with officers of technical committees on research projects begun a year ago was continued, and as a result the Administrative Committee is preparing to submit suggestions to the technical committees for ways in which research activities may properly be stimulated. Special attention is directed to the portion of the Administrative Committee's report discussing the idea of preparing with the aid of the technical committees and publishing a list of researches in the fields of the respective committees that need to be undertaken. An interesting start was made in this direction by Committee D-20 on Plastics through the publication in the January, 1948, issue of the ASTM BULLETIN of a list of needed researches in its field. It is planned to develop this idea in the coming year through discussions with the technical committees.

Two other administrative committees deal with certain phases of our technical activities, namely, the Administrative Committee on Ultimate Consumer Goods and the Administrative Committee on Simulated Service Testing. The annual reports of these two committees appear as Appendices V and VI, respectively, and record briefly the progress made in these respective fields of work. In the field of consumer goods the study begun a year ago of various factors entering into the development of test methods and specifications for such goods is being continued, in addition to which the committee has under study the desirable procedures in the technical committees for the development of standards for consumer goods.

Efforts in the field of simulated service testing contributed to the formation of the new Committee on Structural Sandwich Constructions mentioned below. Other subjects covered in the Administrative Committee's report include studies in the field of bearing metals and lubricants, the establishment of one or more standard humidity tests needed for correlation of laboratory and service tests, and the status of methods used for studying metal deformation and flow, which has led the Administrative Committee to sponsor a symposium on this subject at the annual meeting.

The development of staff contacts with

the work of technical and administrative committees, including the furnishing of secretarial services to administrative committees, has continued during the year. The pace of committee work coupled with the organization in the past two years of several new committees has indicated the need for some expansion in this department of the staff if these services to committees are to be kept as efficient and effective as desired. The Board fully appreciates this need and hopes that such expansion may soon be possible.

New Activities:

A new technical committee was organized in January, 1948, namely, Committee C-19 on Structural Sandwich Constructions. The considerations leading to the organization of this committee were presented rather fully in the Board's report a year ago and it is already clear that there are many important tasks ahead of this committee. At the moment the committee is serving under temporary officers, namely, Prof. A. G. H. Dietz, chairman, D. C. Reid, vice-chairman, and E. C. Hartmann, secretary. The committee is engaged in developing a recommended scope of activities, in the rounding out of a representative personnel and in the setting up of projects and subcommittees. The special study committee on this subject that had advised the Board and officers of the Society in the organization of this new committee has been discharged with thanks for the valued services that it rendered.

On the recommendation of a special conference called for that purpose, the Board has authorized the organization of a new committee E-12 on Appearance Standards to engage in activities along the following lines:

> To improve and develop methods for describing and evaluating the appearance properties (such as color, gloss, opacity, and texture) of engineering materials by:
>
> 1. Formulating terms and definitions generally applicable for describing the appearance of engineering materials;
> 2. Developing, or coordinating with technical committees the development of standard methods of measurement of fundamental and broadly general appearance properties;
> 3. Assisting the technical committees to improve appearance tests by advice and suggestions;
> 4. Reviewing and recommending approval or revision of all A.S.T.M. methods of testing, existing or proposed, involving appearance factors;
> 5. Serving, with the approval of the Board of Directors, as liaison agent between the Society and other organizations in matters concerned with factors of appearance.

M. Rea Paul has been named as chairman *pro tem* to organize this committee, and since the work will involve collaboration with a number of other technical committees a first step has been to invite all interested technical committees to name representatives on the new committee. It is planned to hold a formal organization meeting as soon as a provisional personnel is completed.

As an outgrowth of discussions in the Administrative Committee on Standards there was held at Society headquarters on February 17, 1948, a conference on Acoustical Materials at which a representative group interested in such materials was present. A thorough discussion of the need for tests and specifications for such materials ensued, taking into account the work in and interests of other organizations, particularly the work of ASA Sectional Committee Z24 on Acoustical Measurements and Terminology and of its Subcommittee on Sound Isolation and Sound Absorption Measurements. This conference recommended that work in this field should be undertaken by the Society, the suggested scope to deal with acoustical materials used as airborne sound absorbing materials. This scope would exclude materials in the field of vibration isolation and vibration damping, for which, if work is needed, it might be desirable to consider the establishment of another committee. The Board has approved this recommendation and has authorized the President and Executive Secretary to take the necessary steps to bring about the organization of a new technical committee on Acoustical Materials. The record of the conference has supplied a number of proposals that will greatly facilitate the initiation of this work.

The American Ceramic Society through its White Wares Division and its Board of Trustees has proposed to A.S.T.M. that work in the field of developing test methods and specifications for white wares, which has been handled for many years by the Ceramic Society, be taken over by A.S.T.M. through the formation of an appropriate technical committee. This field includes the various types of sanitary ware as well as tableware. This proposal was accompanied by assurance of support by the Ceramic Society along lines similar to those that have existed for many years in other ceramic fields, notably that of refractories. The Board of Directors has agreed to this proposal and has authorized the President and Executive Secretary, after such further conferences with the American Ceramic Society as may be necessary, to proceed with the formation of a new technical committee on White Wares.

It is appropriate to mention here that the Administrative Committee on Standards is studying the desirability of organizing an A.S.T.M. committee in a closely allied field, namely, that of porcelain enamel. This field would also cover certain types of sanitary ware, but in addition would include porcelain finishes to many articles (such as refrigerators) and certain types of construction materials.

The Board has authorized Committee D-1 on Paint, Varnish, Lacquer, and Related Products to participate in the activities of a proposed joint committee of representatives of certain technical societies interested in paints. Committee D-1 has stated that the work of such a joint committee would be helpful in bringing to the attention of various groups problems connected with technical aspects of paints which could be worked on to the advantage of the industry and of the paint consumer.

The Board of Directors has accepted for the Society a proposal from the Coordinating Research Council (CRC), which is a joint activity of the Society of Automotive Engineers and the American Petroleum Institute, that A.S.T.M. take over from CRC projects relating to maintenance of routine methods for determining combustion characteristics of engine fuels, including services in the certification of reference fuels and the procedure for maintenance of routine testing. This work will be carried on for the Society through Committee D-2 on Petroleum Products and Lubricants, more specifically in its Division on Combustion Characteristics. Administration of certain phases of the work will be done in the headquarters staff. The National Bureau of Standards, through an agreement effective January 1, 1948, has agreed to continue for the Society its services in this connection that have been carried on for some years for CRC. The cost of this work will be reimbursed to the Bureau through the collection of fees for the certification of reference fuels and for participation in nation-wide exchange testing of fuels. The work is done entirely on a non-profit basis and there has been set up for this purpose a special "ASTM-DCC Reference Fuel Account" in the Committee Funds under Committee D-2, disbursements from which are subject to the usual regulations of the Society. An important adjunct to this project has been a new Society publication, namely, "A.S.T.M. Manual of Engine Test Methods for Rating Fuels" which was published in April, 1948, under the immediate sponsorship of Committee D-2.

Committee Scopes and Jurisdiction:

By mutual agreement between the two committees, Committee C-6 on Drain Tile has been formally discharged and its work assigned to Committee C-15 on Manufactured Masonry Units. This has involved the transfer of Specifications for Drain Tile (C 4 - 24) to the latter committee which has formed a suitable subcommittee to take over this work. The official statement of scope of Committee C-15 will be suitably modified to include the subject of drain tile therein. There has been little need for activity in the field of drain tile for a number of years, and it seemed undesirable to continue a technical committee that has had practically no work to do for many years. The effective work of Committee C-6 in the development of the specifications referred to has been recognized by wide acceptance of this specification by both producers and users, and in discharging the committee the Board expressed appreciation of its services.

The scope of Committee C-7 on Lime has been extended to include limestone as used in the chemical, agricultural, and process industries. The revised statement of scope is published in the current Year Book.

The Board has agreed to requests of three committees for modification of scope. In the case of Committee D-2 on Petroleum Products and Lubricants the change in scope is primarily editorial in character, necessitated by extensive reorganization of the division and subcommittee structure of that committee. The revised scope is as follows:

> The promotion of knowledge of petroleum, of petroleum products (including products derived in part from petroleum) and of lubricants; and the recommendation of standards pertinent to these materials.
>
> Standards peculiar to gaseous fuels, road and paving materials, bituminous waterproofing and roofing materials, paint thinners, and electrical insulating materials are excluded from the scope of Committee D-2 and developments in these fields incidental to the normal work of Committee D-2 will be coordinated with the appropriate technical committees of the Society.

In the case of Committee D-19 on Water for Industrial Uses the committee wishes to undertake work on industrial wastes, but not including the subject of stream pollution beyond the effluent from the plant. The Board has accordingly agreed to a change in the committee's title to "Committee D-19 on Industrial Water" under the following revised scope:

> The study and description of industrial water as an engineering material, including terminology; methods of sampling, analysis and testing; interpretation of results of analyses and tests; classification; and the preparation of specifications.

In the case of Committee D-8 on Bituminous Waterproofing and Roofing Materials the scope has been extended to include bituminous siding materials, and a suitable revision of the scope will be published in the next Year Book.

The Board has under consideration the determination of the most suitable place in the Society to undertake work in the development of tests and specifications for flue linings. Discussions are under way with Committees C-15 on Manufactured Masonry Units and C-4 on Clay Pipe.

The Board has considered with Committee E-7 on Radiographic Testing and certain other interested committees the proposal of Committee E-7 to enlarge its scope to include responsibility for all non-destructive testing. This is a field of growing importance, especially in the light of recent developments in magnetic and ultra-sonic testing. While such expansion has been approved in principle, there remain to be worked out procedures of collaboration with other committees of the Society that are directly concerned with non-destructive tests of the materials and products within their scope. It is expected that these questions may be resolved during the coming annual meeting.

During the year jurisdiction for the Standard Specifications for Brazing Solder (B 64) and for Silver Solder (B 73) was transferred from Committee B-2 on Non-Ferrous Metals and Alloys to the Joint Committee on Filler Metal of the A.S.T.M. and American Welding Society. This transfer of jurisdiction was formally approved by the latter Society.

By mutual consent of the two committees concerned, authority for Specifications for Electrodeposited Coatings of Zinc on Steel (A 164) has been transferred from Committee A-5 on Corrosion of Iron and Steel to Committee B-8 on Electrodeposited Metallic Coatings, with the understanding that the latter committee will seek the advice and approval of the former before making any major changes in the specifications and that Committee A-5 will retain jurisdiction over product specifications for electrodeposited coatings on iron and steel. In making this transfer the scope of Committee B-8 has accordingly been revised to close with the following clause: "excluding only specifications for metal products to which electrodeposited coatings are applied."

By mutual agreement between the two committees concerned, jurisdiction over the Tentative Methods for Sampling Molybdenum Salts and Compounds for Metallurgical Use (E 66) has been transferred from Committee E-3 on Chemical Analysis of Metals back to Committee A-9 on Ferro-Alloys. This committee originally wrote the method in question, and it has been decided that it is preferable to have the standard under its jurisdiction.

DISTRICT ACTIVITIES

The report of the Administrative Committee on District Activities, which group supervises and correlates the activities of the A.S.T.M. districts, is appended (Appendix VII). As noted, one of the important accomplishments during the year was the expansion and integration of the areas covered by the districts. This has not only clarified certain district boundaries, but has the added advantage of bringing into the district work a considerable percentage of our members and committee members who previously had not been assigned to a specific district.

The new charter for districts, which vests rather complete autonomy in the district councils, has worked smoothly and much has been accomplished during the year in the interests of the Society. A further amendment of the charter was made during the year to establish a procedure for the organization of new districts. This procedure provides for the appointment by the Board of Directors of councilors *pro tem* and an acting chairman. This group is authorized to organize the new district through the appointment of a nominating committee and election of officers and councilors, as called for in the charter. The Administrative Committee on District Activities conducts this first election.

One new district was formally organized in the early spring of this year, namely the Washington (D. C.) District. Another district centered in the lower Ohio valley around the Cincinnati-Dayton-Louisville-Indianapolis area is in process of organization, and will be the thirteenth district of the Society.

The Northern California District arranged for the participation by the Society in the Pacific Chemical Exposition held in San Francisco in the fall of 1947, and a number of A.S.T.M. members participated in arranging and manning our booth.

The Detroit District Council has constituted the basis for a local committee on arrangements for the 1948 Annual Meeting which has aided greatly in plans for the meeting, the committee being responsible for local housing, entertainment (including ladies' entertainment), dinner and dance, and related matters, and aiding the staff in such things as the Photographic Exhibit.

The Chicago District continued its participation in the Production Conference and arranged for a formal session, thus keeping A.S.T.M. in a prominent position in this area.

Although the President was incapacitated for several weeks and could not attend meetings during this period, he nevertheless visited most of the districts during the year. He and the Executive Secretary visited the Pacific Coast in March, 1948, where both spoke at meetings arranged by the district councils, and held jointly with other societies. One of the important objects of the visit was to perfect arrangements for the national meeting scheduled for San Francisco in October, 1949.

District Meetings:

Each of the districts sponsored technical meetings during the period from July 1, 1947, through May, 1948, and in addition, many of the councils had meetings of their smaller groups to plan district work. Probably the most extensive meeting was the one in Philadelphia in October, 1947, comprising a Symposium on Flammability followed by a dinner and an evening session on Textile Fibers. The papers and the discussion at the Detroit meeting in October, 1947, on Gasoline and Lubricating Oils were published. One of the most interesting meetings was that in New York in February featured by a discussion on Growing Crystals for Communication Purposes. Two meetings of very broad interest, and at which there was good representation from the ladies, were the one in New York on Electronic Cooking and the April meeting of the Pittsburgh District under the title "Japan Through the Eyes of a Metallurgist."

President Boyd presented his paper on "Research—Everybody's Doing It Now" at several district meetings, including the first one held in the Ohio Valley at Dayton. Some details of district meetings are given in the accompanying table.

One aspect of these district meetings should be noted, namely, that several have been held jointly with local sections or branches of other national societies and for every meeting, whether joint or under sole district sponsorship, invitations have been extended to groups of engineer members of other sections.

NATIONAL MEETINGS

A very extensive technical program featured the 1947 Annual Meeting in Atlantic City, and numerous publications comprising the symposiums and papers presented at the meeting have been issued.

Since this was the Society's Fiftieth Annual Meeting, special cognizance was taken of this event at the Annual Meeting dinner. President Carpenter referred to the great progress made by A.S.T.M. since its forerunner, the American Section of the International Association for Testing Materials, was organized in 1898.

During A.S.T.M. Committee Week

A.S.T.M. MEETINGS JUNE, 1947-MAY, 1948, INCLUSIVE.

Meeting	Place and Date	Notes
Annual Meeting	Atlantic City, N. J. Chalfonte-Haddon Hall June 16-20, 1947	19 Technical Sessions 350 Committee Meetings (1786 in attendance)
A.S.T.M. Committee Week	Washington, D. C. Hotels Statler, Shoreham, Wardman Park and others March 1-5, 1948	Over 300 Committee Meetings (about 1100 in attendance)
Spring Meeting	There was no Spring Meeting held in 1948.	
District or Local (sponsored by Councils indicated)	Detroit, Mich. Rackham Memorial Building October 8, 1947	Petroleum Lubricating Oils as Related to Automotive Equipment, by C. M. Larson; The Efficient Production and Utilization of Motor Gasoline, by W. M. Holaday.[a] (320 in attendance)
	Philadelphia, Pa. Edison Building Auditorium October 16, 1947	Symposium on Flammability[b] (afternoon) with three technical papers (by Messrs. Frederic Bonnet, R. W. Little, and C. W. Dorn); a dinner with an address on Textiles for Peace and War,[c] by Col. S. J. Kennedy; and an evening session on Textile Fibers, with three papers, by Messrs. B. L. Whittier, D. H. Powers[c], and Rene Bouvet. (200 in attendance)
	Providence, R. I. (New England) Providence Engineering Society October 30, 1947	Woven Narrow Fabric and Its Relation to the Textile Industry, by E. J. Gibbons; Braiding Narrow Fabric and Its Relation to the Textile Industry, by F. W. Fraim; New Methods and Techniques of Testing Textile Materials, by E. R. Schwarz. (150 in attendance)
	New York, N. Y. Engineering Societies Bldg. October 30, 1947	Lecture and Demonstration on Silicones by Kingsley W. Given. (300 in attendance)
	Pittsburgh, Pa. Mellon Institute Auditorium October 31, 1947	High-Strength Steels, by H. Malcolm Priest. (65 in attendance)
	Buffalo, N. Y. (Western New York-Ontario) Hotel Lafayette November 18, 1947	Paint—the Servant of Industry, by W. H. Lutz; Recent Development in Portland Cement,[e] by J. F. Barton. (50 in attendance)
	Los Angeles, Calif. (Southern California) Rodger Young Auditorium December 8, 1947	A.S.T.M. Specifications in Building Construction, by Virgil Rapp; Applications of Aluminum in Building Construction, by F. L. Landon. (90 in attendance)
	New York, N. Y. Engineering Societies Bldg. December 11, 1947	Low and High Frequency Electronic Induction Heaters, with Technical Sound Film and Demonstration of Electronic Cooking, by C. B. Hart. (150 in attendance)
	Philadelphia, Pa. Benjamin Franklin Hotel February 3, 1948	When Metal Breaks—From the Practical Standpoint, by O. J. Horger; From the Theoretical Standpoint, by J. H. Holloman. (200 in attendance)
	New York, N. Y. Engineering Societies Bldg. February 17, 1948	Growing Crystals for Communication Purposes, by A. C. Walker; Research in Action, by J. R. Townsend. (150 in attendance)
	Philadelphia, Pa. Franklin Institute February 24, 1948	Petroleum Products—Production, Consumption and Research; T. A. Boyd (on the importance and benefits of research)[f]; and E. T. Knight (on the supply situation). (125 in attendance)
	Dayton, Ohio (Lower Ohio Valley) Dayton Engineers Club February 26, 1948	First Lower Ohio Valley Technical Meeting. Research—Everybody's Doing It Now,[f] by T. A. Boyd; Case Histories of Research, by R. Burns, J. M. Purdy, and H. K. Nason. (135 in attendance)

A.S.T.M. MEETINGS JUNE, 1947–MAY, 1948, INCLUSIVE.—Continued.

Meeting	Place and Date	Notes
District or Local (sponsored by Councils indicated)	Los Angeles, Calif. (Southern California) Rodger Young Auditorium March 18, 1948	Jointly with S.A.E. Research on Materials and Mechanisms,[f] by T. A. Boyd; Research and Standardization in A.S.T.M. by C. L. Warwick. (150 in attendance)
	Chicago, Ill. Hotel Stevens March 23, 1948	A technical panel, part of the Chicago Technical Societies Conference. The A.S.T.M. session covered "The Value of Specifications in Production," with three papers:[g] Buying Steel by Hardenability and by Composition Specifications, by Harry B. Knolton; Development of Modern Alloys for Die Casting, by Donald L. Colwell; Selection and Control of Plastics, by Harvey A. Anderson. (105 in attendance)
	San Francisco, Calif. (Northern California) Engineers Club March 25, 1948	Jointly with A.S.M.E. Research on Materials and Mechanisms,[f] by T. A. Boyd; Research and Standardization in A.S.T.M. by C. L. Warwick. (135 in attendance)
	Cleveland, Ohio Cleveland Engineering Society April 7, 1948	Research—Everybody's Doing It Now, by T. A. Boyd; Value of Research in Industry, by C. F. Prutton. (150 in attendance)
	St. Louis, Mo. Engineers Club April 9, 1948	Research—Everybody's Doing It Now,[f] by T. A Boyd; Case Histories of Research, by F. J Curtis. (100 in attendance)
	New York, N. Y. Engineering Societies Bldg. April 13, 1948	Weather: Modification of Clouds in the Free Atmosphere, by V. J. Schaefer; Margin of Error in Weather, by P. E. Kraght. (200 in attendance)
	Boston, Mass. (New England) Northeastern University April 14, 1948	Research—Everybody's Doing It Now, by T. A. Boyd; Finding Answers to Materials Problems: Plastics, A.G.H. Dietz; Electrical Equipment, A. L. Shields. (65 in attendance)
	Pittsburgh, Pa. Mellon Institute Auditorium April 26, 1948	Japan Through the Eyes of a Metallurgist—Ferrous Metals, E. T. Barron; Non-Ferrous Metals, W. W. Wentz. (150 in attendance)
	Niagara Falls, Ont., Canada (Western New York-Ontario) Red Casque Inn May 27, 1948	Jointly with Niagara Peninsula Branch, Engineering Institute of Canada. Materials for Gas Turbines, by R. B. Gordon. (115 in attendance)

---

[a] Papers and discussion published as separate A.S.T.M. Publication STP 81, issued February, 1948.
[b] Symposium on Flammability published in ASTM BULLETIN No. 150, January, 1948.
[c] Extended abstracts published in ASTM BULLETIN No. 149, December, 1947.
[d] Extended abstract published in ASTM BULLETIN No. 150, January, 1948.
[e] Extended abstracts published in ASTM BULLETIN No. 150, January, 1948.
[f] Modified version published in ASTM BULLETIN No. 152, May, 1948.
[g] Condensed abstracts published in ASTM BULLETIN, No. 152, May, 1948.

held in Washington in March, 1948, there were meetings of 300 of the Society's technical committees and subgroups. This year there was no Spring Meeting with the usual technical sessions during Committee Week.

1949 Meetings:

The 1949 Annual Meeting will be held in Chalfonte-Haddon Hall, Atlantic City, over the dates June 27–July 1. Of particular interest is the decision to hold a National Meeting of the Society on the Pacific Coast during 1949. San Francisco will be the city and the dates are October 10 to 14. Sessions will be held in the Fairmont Hotel. The May BULLETIN gives preliminary announcement of the plans.

Committee Week and the Spring Meeting in 1949 will be held in Chicago during the week beginning February 28, at the Edgewater Beach Hotel.

PUBLICATIONS

Our publications are in a real sense the "product" of the Society's work and constitute a most important service to the members. The editorial and publication activities of the staff loom large in A.S.T.M. affairs and place considerable responsibility in the hands of the Administrative Committee on Papers and Publications which guides and directs those activities for the Board. Therefore, the report of that committee, given in Appendix IV, covers a most important phase of A.S.T.M. work and should be of interest to a great many members. The report contains important recommendations concerning the methods of issuing our major publications, and also gives interesting information about the publications of the past year as well as plans for some future publications.

The annual volume of *Proceedings* (1206 pages) and the five Supplements to the Book of Standards (2015 pages) were the major 1947 publications. The ASTM BULLETIN (6 issues, 668 pages) continues to grow and to be used to an increasing extent for the publication of certain types of technical papers. Twelve compilations of standards were printed during the year. These and the various Special Technical Publications listed in the committee's report constituted an important part of the Society's 1947 publication program.

Publication Policies for the Future:

The Board has felt for several years that the publication structure of the Society should be re-examined and studied in the light of recent developments, particularly the increase in technical papers and the steady growth of standards. The use of the BULLETIN for technical papers, begun on a major scale about ten years ago, led to suggestions that possibly the BULLETIN should be expanded into a monthly technical journal, in which *all* technical papers (including those presented at the annual meeting) and committee reports should be published, eliminating the bound volume of *Proceedings* except as members might order a yearly binding of the sections of the journal devoted to papers and reports. An alternate suggestion was to publish the technical papers in a monthly or quarterly book of *Proceedings*, retaining the BULLETIN essentially as a news medium. One of the principal ideas back of these suggestions was that of "current" rather than "annual" publications of A.S.T.M. papers. As to publication of standards, the increasing volume of standards and the utility and popularity of the separate compilations of standards led to various suggestions, among them the use of the compilations in place of the Book of Standards as the main avenue of publication, and the use of loose-leaf or loose-unit methods of publishing. At the request of the Board, all these questions have been thoroughly studied by the Administrative Committee on Papers and Publications over a two-year period and the committee's report thereon with recommendations appears in Appendix IV.

The Board of Directors has approved these recommendations and adopted the publication policies that they constitute. These are briefly summarized as follows:

As to Technical Papers.—No major change will be made. Preprinting of papers during the year, and before and after the annual meeting, will bring technical data to the members as rapidly as possible. The bound volume of *Proceedings* will be continued to provide a permanent record, the need for and value of which is clearly established by these studies. Although the proposal to publish a monthly technical journal is not favored, the BULLETIN will be expanded from six to eight issues annually, to provide increased facilities for timely publication of papers of current and general interest and opportunity for more frequent contact with members and other readers of that publication. The staff has been directed to proceed with plans for this important expansion.

As to Committee Reports.—These will

be handled substantially as heretofore, with increased use of the BULLETIN for progress reports. The Board has deferred action on the recommendation that committee reports be published separately from the technical papers in a paper bound volume, possibly distributed to members only on request, pending opportunity for further consideration in the light of a proposal that changes be made in the Society's procedures for receiving and acting upon committee reports. Brief reference to this proposal appears later in this report.

As to Standards.—The loose-leaf and loose-unit systems of publication of the Book of Standards have been rejected as not practicable. The advantages that are usually cited for such methods of publication cease to exist when, as is the case with A.S.T.M. standards, as much as one-fourth of the body of standards may be revised annually; and the disadvantages—including that of increased costs—are considered to be so weighty as to warrant no further consideration of such methods. Beginning with the 1949 edition, the Book of Standards will be published in six parts (for an approximate classification of contents, see Appendix IV) and—as a measure of economy and speeding up the appearance of both the Book itself and many of the separate compilations of standards—the Book will be published as a grouping together of the compilations, by making up and printing the individual compilations and overrunning on press the desired number of impressions for the bound volumes of the Book of Standards. Special methods of paging and indexing the Book of Standards will be required. This combination method of printing cannot, of course, be used for publishing the Supplements to the Book of Standards.

The Board now has under consideration the effect of issuing the Book of Standards in six parts on the current methods of distributing the Book and its Supplements to the members on the present five-part basis.

ADMINISTRATIVE MATTERS

Amendment of By-laws in 1947:

As a matter of official record, the Board of Directors confirms the announcement in the December, 1947, BULLETIN that the amendments of the By-laws referred to letter ballot of the Society at the last annual meeting were adopted by the following votes:

Amendment of Article III, Section 2, Nomination of Officers:

Legal ballots received	952
Deduct ballots marked "Not Voting"	58
Number of votes cast	894
Affirmative	890
Negative	4

Amendment of Article VIII, Section 1, Increase in Dues:

Legal ballots received	952
Deduct ballots marked "Not Voting"	47
Number of votes cast	905
Affirmative	849
Negative	56

Revision of Regulations Governing Technical Committees:

In order to provide suitable authorization for the technical committees to raise funds in support of certain committee expenses, such as compensation for technical or administrative assistants, certain costs of meetings, and certain travel expenses when duly authorized, Sections 28 and 29 of Article VII, Expenses of Technical Committees, of the Regulations Governing Technical Committees have been revised during the year in the manner provided, namely, by the Board with the advice of the chairmen of technical committees. At the same time, several minor editorial changes were made in other sections of that Article. The Article as now revised and in effect is as follows:

VII. EXPENSES OF TECHNICAL COMMITTEES

25. Current Expenses.—Expenses for postage incurred in connection with the business of committees will be refunded by the Society on vouchers approved by the chairmen of these committees.

26. Stationery.—Correspondence relating to the business of committees or subcommittees shall be conducted on official stationery which will be furnished by the Society.

27. Extraordinary Expenses.—Expenses for items other than postage will not be assumed by the Society, unless such expenditures were incurred in pursuance of previous authorization of the Board of Directors, on recommendation of the chairman of the committee concerned, and within amounts specifically fixed by the Board of Directors.

28. Special and Research Funds.—Committees may raise special funds through voluntary contributions from their members and voluntary registration fees at committee meetings, or through other means authorized by the Board of Directors, for the purpose of defraying such expenses as salaries or special compensation for technical or administrative assistants, costs of committee meetings, and such travel or other expenses of committee officers as may be authorized by the committee and approved by the Board of Directors. Committees are also authorized to raise funds in support of research. All such special and research funds thus collected, unless otherwise authorized by the Board of Directors, shall be deposited with the Society and placed to the credit of the committees on the books of the Society, subject to disbursement by the Executive Secretary only on vouchers signed by the chairman of the committee concerned.

Payment of voluntary contributions or registration fees as authorized in this section shall not be required as a condition of committee membership, committee meeting attendance, or participation in committee work.

29. Salaries and Fees.—Committees shall not be authorized to pay salaries or professional fees in any form to any of their officers or members. Technical or administrative assistants to a committee, or assistants in connection with research work, may be engaged at salaries or special compensation fixed by the committees concerned, provided that funds for such salaries or compensation shall previously have been deposited with the Society. Payments for such purposes shall be made by the Executive Secretary of the Society only on vouchers approved by the chairman of the committee concerned.

Presentation of Committee Reports:

A two years' trial of the plan of presenting committee reports at the annual meeting at sessions devoted exclusively to such reports has clearly indicated that such sessions are not successful, and at this annual meeting the earlier procedure of presenting committee reports at the general technical sessions is being resumed. Consideration of this matter has opened up the whole question of Society procedure in receiving committee reports and in acting upon recommendations for standards and tentatives. What might be termed "the annual meeting procedure" and the procedure between annual meetings for consideration by the Administrative Committee on Standards of reports on tentatives, have been examined in relation to the needs imposed by the growth of committee work and the increasing extent to which not only the actual writing of specifications and methods of tests but essential responsibility and almost final authority for the committee are of necessity being delegated to subcommittees and divisions of the main technical committees. As a result, the Board is considering and will confer with the technical committees regarding a plan for establishment of review panels to act for the Society during annual meetings in receiving committee reports much as the Administrative Committee on Standards acts for the Society in the interim between meetings.

Administrative Organization:

The administrative organization set up two years ago when the new Board of Directors took office, and which was rather fully described in the report a year ago, has proved to be quite satisfactory. The various administrative committees handle many important affairs of the Society, reporting thereon to the Board, as will be seen from their reports appended hereto. A second year of work by the Executive Committee of the Board has confirmed the belief that much necessary routine business of the Board can be handled in meetings of the smaller Executive Committee, with the result that the Board in its three meetings since last June has been able to give adequate time to the consideration of a number of important questions of administrative and operating policy.

During the year there were two additions to the stenographic and clerical staff, bringing the total staff personnel to 46, of whom 11 are on the administrative and technical staff and 35 on the stenographic and clerical staff. In addition several clerks and stenographers are employed from time to time on a part-time basis to meet peak load conditions.

The Board has under study a report of the Executive Secretary on the needs for additions to the staff, which include three men for the administrative and technical staff and eight additions to the stenographic and clerical staff. While all of the proposed expansion is believed to be justified, it is neither possible nor necessary to put it at once into effect. However, it is now possible to make a start and the Board has authorized the Executive Secretary to add a youngman to the administrative staff to work on development of membership and publication sales under the direction of the Assistant Secretary; also an additional stenographer in that department and an additional bookkeeper in the finance department. It has been quite evident in the past few years that more intensive work in acquiring new members and in promotion of publication sales and advertising would strengthen the Society's work. In the long run the cost of additions to the staff for such work should be more than repaid through increased income.

Society Headquarters:

It is very gratifying to be able to report that the Society Headquarters continue to be well adapted to the needs of the staff and that the facilities provided for the members and committees of the Society are being used to an increasing extent. Many committee meetings and conferences have been held in the Society rooms during this past year, and the members and committees are urged to take fullest advantage of these facilities. In particular the staff will gladly do all it can to facilitate the holding of successful committee meetings at Headquarters.

After considerable delay, the alterations to the building were completed early in 1948 with the installation of the air-conditioning units, which are so constructed as to be used for both heating and cooling the building.

Financial Aspects.—The total cost of the Headquarters, including purchase of land and building and alterations, will be about $184,000. The total of contributions to the Building Fund up to June 1, 1948, is $165,640, which represents additional contributions received through the year of close to $9600. Of this amount approximately $6800 has been raised by the technical committees of the Society and the Board is most appreciative of the interest that the committees have shown and of the substantial contributions that the committee members in the aggregate have made.

Other additions to the Fund, principally interest and profit from sale of investments, have added to the assets so that at the moment the deficit between cost and receipts in the Building Fund is in the neighborhood of $17,500. The Board would like to see the Society own the headquarters building completely free of indebtedness and invites further contributions from committees and members who desire to aid in attaining this goal.

MEMBERSHIP

On June 1, 1948, the membership of the Society totaled 6438, the highest figure yet reached, compared with a total of 6313 on June 1 one year ago. Statistics showing gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1947	June 1, 1948	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	14	16	...	..	1	..	3	..	1	3	2
Perpetuity and Life	12	12	...	..	..	..	..	..	..	...	..
Sustaining	213	211	2	..	..	5	1	4	7	5	2[a]
Company, Firm, etc.	1650	1654	97	15	..	22	11	127	134	138	4
Individual, etc.	4305	4435	254	76	43	9	42	470	382	512	130
Junior	119	110	14	17	..	21	..	43	52	43	9[a]
Total	6313	6438	367	108	44	57	57	644	576	701	125
Student	336	577	15	136	..	22	.	414	173	414	241

[a] Net loss.

While the net gain for the year, 125, was considerably less than for the past several years, the Board of Directors feels that this situation might be expected, in part due to a full return to peace-time activities and a consequent dropping of memberships held only during the war period, plus the impact of increased dues, especially for companies and sustaining members. Nevertheless, an analysis of the resignations would indicate that only a moderate number are attributable to increased dues, the greater percentage being influenced by normal causes which arise from year to year. It is significant to point out that the number of new members, 644, is only slightly below that of the previous year, which was the highest in the Society's history, 673.

Sustaining Members:

There have been four new sustaining members acquired, and there were seven losses, all but two of these being transfers to either company or individual membership. The four new sustaining members are:

Cluett, Peabody and Co., Inc.
Eastman Kodak Co.
Ford Motor Co.
Mueller Brass Co.

Honorary Memberships:

The Board of Directors has elected to Honorary Membership in the Society the following three members: Harvey L. Curtis, Consulting Physicist and formerly Principal Physicist, National Bureau of Standards, Washington, D. C.; Frank H. Jackson, Principal Engineer of Tests, Public Roads Administration, Washington, D. C.; and William B. Price, for many years Chief Chemist and Metallurgist, Scovill Manufacturing Co., Waterbury, Conn., now retired. They will be presented to the Society for formal award of honorary membership during this Annual Meeting.

40-Year Members:

Since the plan of awarding certificates commemorating forty years of continuous membership in the Society was established, 115 such certificates have thus far been awarded and this year the following 23 companies and individuals have qualified:

H. Austill
Brown University, Division of Engineering
Allan W. Carpenter*
Columbia Steel and Shafting Co.
Crane Co.
Roland P. Davis
Harold S. Falk
Henry A. Gardner
General Electric Co.
Prevost Hubbard
International Paper Co.
Albert J. Loepsinger
Frank M. Masters
Lesley McCreath
Charles E. Paul
Pecora Paint Co.
Alfred P. Poorman
Purdue University Library
Joseph T. Ryerson & Son, Inc.
H. H. Scofield
Union Drawn Steel Division, Republic Steel Corp.
United States Pipe and Foundry Co.
Alan Wood, 3d

---

* Deceased, May 1948.

Deaths:

The Society has lost the following 44 members through death:

	Date of Membership
Archer, Merton T.	1915
Bampfield, A. E.	1945
Barbour, Frank A.	1898
Boyd, J. E.	1937
Carpenter, Allan W.	1908
Clements, Frank Orville	1905
Conradi, L. C.	1928
Corley, Ralph A.	1936
Crowley, Dennis A.	1940
Dalton, William E.	1936
Evans, Robert G. N.	1943
Fisher, T. W.	1944
Gagnon, Joseph G.	1935
Granger, Robert R.	1946
Henderson, C. L.	1925
Herron, James H.	1911
Hodge, W. B.	1936
Hughes, C. A.	1945
Jablonsky, Roy	1944
Kellogg, C. A.	1925
Kempf, Louis W.	1935
Kendall, Earl H.	1940
Kirschbraun, Lester	1909
Krogh, A. T.	1939
Martin, Stewart	1946
McMullen, E. W.	1916
Mulholland, L. F.	1922
Openshaw, J. Edward	1938
Page, Carlisle W. C.	1944
Pardee, Powell	1912
Pearson, J. C.	1918
Power, L. E.	1927
Primrose, John Stewart Glen	1917
Ramage, John H.	1936
Roby, L. Edward	1931
Russell, David A.	1932
Sawyer, J. S.	1937
Slack, Fred F.	1938
Smith, Howard Agnew	1942
Sullivan, Charles L., Jr.	1917
Suter, Alfred	1916
Wagner, Ernest J.	1943
Washburn, D. E.	1925
Young, John M.	1908

In addition to the above, the following representatives of company members of the Society passed away:

Laurance Bowen, Pecora Paint Co.
Samuel B. Bowen, Jr., Pecora Paint Co.
H. G. Elledge, Diamond Alkali Co.
Howard W. Gilbert, National Malleable and Steel Castings Co.
Stanley M. Hands, City of Oakland, Calif., Engineering Lab., Street Dept.
F. A. Kartak, Marquette University
Robert B. Lewis, Tinius Olsen Testing Machine Co.
Joseph Mattiello, Hilo Varnish Corp.
Benjamin L. McCarthy, Wickwire Spencer Steel Division, The Colorado Fuel and Iron Corp.
C. R. Palmer, Palmer Thermometers, Inc.
G. G. Powell, City of Toronto, Ont., Canada, Department of Works.
W. W. Troxell, The Glenn L. Martin Co.
H. E. Zumbrun, Morehouse Machine Co.

Several of these men had been affiliated with the Society continuously for many years and rendered outstanding service in advancing the Society's purposes, particularly through work on its technical committees. Dr. F. O. Clements, a Past-President and for many years Technical Director of the General Motors Research Laboratories, was a loyal supporter of the Society and stimulated much interest in our work, especially in the Detroit area. The esteem in which he was held is indicated by the fact that he was an Honorary Member. Messrs. Frank A. Barbour, E. W. McMullen, and J. C. Pearson had served terms as members of the Society's Board of Directors and had participated extensively in technical committee activities. Mr. McMullen was especially active in the work of Committee D-1 on Paint, Varnish, Lacquer and Related Products. Mr. Pearson was an authority in the field of cement and concrete and did much to stimulate the presentation of suitable papers and reports in these fields. L. C. Conradi had been active in several technical groups and was a former Chairman of Committee D-11 on Rubber Products. Dr. J. H. Herron, a long-time member, took an intensive interest in A.S.T.M. and aided greatly in the work of the Cleveland District, of which he was a former chairman. Allan W. Carpenter, likewise a long-time member, was active for many years in Committee A-1 on Steel. E. H. Kendall, an authority in the field of materials for distribution of electrical energy, did outstanding work in several committees, notably as Secretary of Committee B-1 on Wires for Electrical Conductors. Alfred Suter, an authority on the testing of textiles and a notable designer of testing machines and related equipment, was a highly respected and active member of Committee D-13. J. J. Mattiello had a distinguished career in the paint field, was an active member of Committee D-1, and in 1946 presented the Edgar Marburg Lecture.

Membership Comments:

Reference should be made to the very active interest and work of the Committee on Membership, headed by Carlton H. Rose. Under the guidance and stimulation of this committee several membership promotional activities have been carried out during the year and others are in prospect. A special mailing to the entire membership resulted in the names of several hundred companies and individuals who were prospective members, and the invitations extended have been quite productive of new members. Letters to individuals and companies who had requested that their membership be relinquished resulted in keeping a number of these in the Society through a different class of membership.

The Membership Committee plans to continue its stimulation of membership promotional work in the technical committees and also in the various districts. The latter can be most helpful in follow-up work.

Among the several projects on which the Membership Committee is working is a survey of certain industrial fields to determine whether our membership could not be extended considerably in those fields.

FINANCES

Report for the Fiscal Year 1947:

The annual statement of the finances of the Society is presented in the form of the report of the auditors for the fiscal year January 1 to December 31, 1947, which appears in Appendix I. The report gives the balance sheet of assets and liabilities, including general funds, building fund, and other special and designated funds; a statement of receipts and disbursements classified into "operating" (budgeted) and "nonoperating"; details of the building fund and of special funds; and investments of Society funds.

Total operating receipts were $428,063.92, the largest in the Society's history. Of this amount receipts from dues and entrance fees were $144,430.52 or 33.7 per cent; receipts from sales of publications were $251,708.43 or 58.8 per cent; receipts from miscellaneous sources consisting principally of advertising, interest and dividends, and registration fees, totaled $31,924.97 or 7.5 per cent. With expansion of Society activities the volume of publications for sale has naturally increased and has provided an increasingly large proportion of total income. Approximately 80 per cent of publication sales are accounted for by publications growing out of our standardization work, the remaining 20 per cent representing sales of Proceedings, technical papers, Bulletin, and many special and miscellaneous publications.

Operating disbursements with all current bills paid (as detailed in the auditors' report) and including $20,000 placed in the reserve for the next Book of Standards were $430,396.52. Of this amount $195,616.95 (including the above-mentioned reserve) was spent for publications and $144,600.57 for salaries. These two items always constitute the major operating expenses, being for 1947 about 80 per cent of the total. The disbursements for salaries are naturally the largest in the Society's history, providing as they do for four additions to the staff during the year and general salary increases effective at the beginning of 1947. Notwithstanding, the percentage ratio of disbursements for salaries to total disbursements, namely, 33.6, is the lowest for a number of years. The remaining disbursements of $90,179.00 were divided among the items of general office expense, expense of technical committees and district councils, meetings, headquarters occupancy (equivalent rent) and miscellaneous items.

The difference between operating receipts and disbursements constitutes an operating deficit of $2322.60, which is about 0.5 per cent of receipts. This deficit has been met by drawing from certain reserve funds.

In view of this operating deficit the usual payment into the principal of the A.S.T.M. Research Fund of one-half of the current entrance fees or about $2700 was deferred.

The auditors' report includes a statement of the financial position of the Society for the past five years. The surplus in the General Funds account on December 31, 1947, was $157,418.09, compared to $144,210.75 a year ago. The ratio of surplus to actual operating disbursements at the current level is approximately 36 per cent. Expressed in another way, the surplus is equivalent to only four months' operating expenses, which the Board of Directors feels is much too low a ratio for sound operation.

The auditors' report also includes a statement of cash receipts and disbursements in the Building Fund as well as a balance sheet for the Fund as of December 31, 1947. As of that date the alterations to the headquarters building had not been completed so that the building and construction costs given in the report, namely, $148,865.26 are not final costs. The final cost will be approximately $153,750 and will be shown in the next auditors' report. In the meantime, as noted in the current report, there was paid into the Building Fund from the General Funds of the Society the sum of $3200 as reserve for depreciation, this constituting a charge in the general budget against headquarters occupancy expense.

Investments of Society Funds as of December 31, 1947, are given in the auditors' report, both book and market values being shown. As of that date, the book value of all investments was $240,300.91; the market value of these investments was $228,746.49, a depreciation of approximately 5 per cent. On the basis of market values, 55.5 per cent of investments were in bonds, 10.2 per cent in preferred stocks, and 34.3 per cent in common stocks; government bonds constituted 45.7 per cent of all securities.

The following changes during the fiscal year in the investment portfolio are noted:

General Funds

Bought	Cost
10 shares American Cyanamid Co. Cum. Pfd. A 3½'s	$1 042.53
50 shares Montgomery Ward and Co. Common	1 813.99
10 shares Texas Co. Common	450.00
	$3 306.52

Research Fund

Bought	
20 shares Montgomery Ward and Co. Common	$1 209.33
16 shares U. S. Gypsum Co. Common	960.00
	$2 169.33

Building Fund

Sold	Received
$10,000 U. S. Treasury Bonds 1967–72, 2½'s	$10 321.88

Sam Tour Award

Bought	Cost
$1500 U. S. Defense Savings Bonds "G"	$1 500.00

The profit on sale of the Treasury Bonds in the Building Fund was $165.68.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of May 1, 1948, may be summarized as follows:

Year Book (1947)	1 227
Proceedings (All back copies and incl. 1947)	4 394
Index to Proceedings	1 089
1946 Book A.S.T.M. Standards	
Part I A	855
Part I B	1 638
Part II	1 678
Part III A	944
Part III B	1 175
1947 Supplements to Book of Standards	
Part I A	3 150
Part I B	2 072
Part II	2 202
Part III A	2 386
Part III B	2 119
A.S.T.M. Methods of Chemical Analysis	1 694
Compilation of Standards	14 119
Selected Standards for Students	34 438
Symposiums and Special Reprints	62 024
Reprints of Standards (approximate)	145 000
Index to A.S.T.M. Standards (1946)	129
Radiographic Standards	62

1948 Finances:

In approaching the problem of budgeting the Society's operations for 1948 the Board first established the policy that current operations must be carried on within the income for the year; in other words, there would be no "deficit financing." Deficits in the two preceding years have amounted to slightly over $30,000, brought about as is well understood through the greatly increased costs of operation, especially printing costs, and this situation was a fundamental reason for increasing the membership dues effective January 1. This increase itself posed the problem of estimating income from dues under the new scale. There had been such universal support of the new dues by the membership at large that it was felt losses from resignations due to the increase would not be excessive, and after careful analysis the estimated receipts from dues and entrance fees were fixed at $200,000, which is approximately $56,000 higher than for the preceding year or an increase of about 40 per cent.

Estimates of publication sales were fixed at $221,000 and receipts from miscellaneous sources at $43,000, the latter including such items as advertising, interest and dividends, registration and other fees, and income from the A.S.T.M. Exhibit. These figures led to an estimate of current receipts of $464,000.

In fixing the budget of disbursements full account was taken of the requirements for salary increases for the staff and for the normal publication schedule, with allowance for such increases in printing and other costs as could be foreseen and anticipated. Provision was then made for all the other normal expenses of operation, with the understanding that because of the considerable uncertainty of the budget of receipts there would be no expansion of publication or other services and no major commitments for staff expansion until the situation for 1948 was more clearly known and some reasonable estimate could be made of operations for 1949. This estimate and line of reasoning led to a budget of disbursements of $437,000, leaving an estimated favorable balance between receipts and disbursements of $27,000 to be set aside as a current reserve and contingency fund.

The Board is pleased to report to the

Society that the first five months' operations under this budget are on the favorable side; receipts in all categories are up to expectations for the five-month period and there have as yet been no substantial increases in printing costs that had not already been anticipated. In view of this situation the Board has authorized a modest expansion of staff, which is referred to elsewhere in the report.

To complete reference to 1948 finances thus far this year, it is noted that during the first quarter 19 shares of American Cyanamid Co. 5 per cent cumulative preferred stock (book value $190) were called netting $200.30, and there was also purchased for account of the Book of Standards Reserve $20,000 U. S. Treasury Bonds 2's, 1949–51 at a cost of $20,275. Late in May the following common stocks were sold:

From General Funds: 100 shares Atlantic Refining Co., 100 shares S. S. Kresge Co., 100 shares Mathieson Chemical Corp., 75 shares J. C. Penney Co., 60 shares Texas Co., and 50 shares U. S. Gypsum Co.

From the Research Fund: 100 shares Ohio Oil Co. and 16 shares U. S. Gypsum Co.

The book value of these securities was $20,005.01; the net proceeds from their sale was $29,591.54, or a profit in the transaction of $9586.53. At the date of writing this report the proceeds of this transaction have not yet been received from the brokers and are therefore not reflected in the records of the Society.

The total book value of Society investments as of May 31, 1948 in all funds was $260,385.91, which will be reduced to $240,380.90 when effect is given to the above-mentioned sale. The Society's cash balance in General Funds as of the same date was $187,274.24, which will be increased by the proceeds of the sale when received.

RETIREMENT PLAN FOR EMPLOYEES

During the year two employees, out of six newly eligible, joined the Retirement Plan and there was one loss through resignation from the staff, bringing the number of participants to 25 out of 38 eligible employees. All of the men on the staff are participating, and most of the 13 women who have not joined the Plan are new and younger employees. In addition, new retirement policies were taken out for five employees to bring the income from their retirement policies closer to their specified retirement income, which it will be recalled is determined by the formula: One per cent for each year of service, times an average salary based on the five highest years, except that no portion of any salary in excess of $5000 per year is included in arriving at the retirement income.

The Society's total contribution in premium deposits for retirement policies for the above-mentioned 25 employees is at the annual rate of approximately $11,275, which is roughly $7\frac{1}{4}$ per cent of the present salary roll and $2\frac{1}{2}$ per cent of estimated current disbursements. The total of employees' annual contributions, including insurance benefits which most of them have elected, is approximately $3000.

Provisions of the Plan were amended slightly during the year to provide that henceforth new employees will be eligible for participation upon completion of one year's service, provided they are under age 55 for men and under age 50 for women when employed. Heretofore there has been no upper age limit for admission to the Plan. Furthermore, interpretations of the Plan have been added which clarify the manner in which the Plan will operate for a participating employee who, under certain conditions, (1) may be retired earlier than his or her normal retirement date or (2) may be retained in the Society's employ beyond such normal retirement date.

This year marks the tenth anniversary of the institution of the Retirement Plan. In these first ten years the Society has put into the Plan approximately $42,115 and the employees have put in approximately $16,775, or a total of nearly $59,000. These payments or premium deposits—together, of course, with future annual deposits as required—provide for the 25 participants retirement income policies having an aggregate guaranteed value, exclusive of dividends, at maturity (age 65 for men, age 60 for women) of $351,500, or an *average* fund of $14,060 per participating employee. Admittedly aggregate totals and averages based thereon do not portray the practical results in so far as individual employees are concerned, unless it is added that the maximum retirement income provided to any individual employee now stands at $195 per month and the minimum provided is now $20 per month.

COOPERATIVE ACTIVITIES

Effective cooperation with the Federal Specifications Board and its various technical committees has been continued during the year. Contacts at the staff level are maintained which have served to keep the staffs and through them the committees of the two organizations generally informed of important developments in the writing of specifications. Many Federal specifications in draft form are reviewed by committees of the Society and comments submitted to the Federal Specifications Board for its consideration, thus serving as a further means of bringing Federal and A.S.T.M. specifications more closely together.

The Industry Advisory Council, which serves in an advisory capacity to the Federal Specifications Board and upon which the Executive Secretary represents the A.S.T.M., held one meeting during the year jointly with the Federal Specifications Board and the U. S. Standards Commodity Catalog Board.

The A.S.T.M. Ordnance Advisory Committee formed on the invitation of the Ordnance Department of the Army was organized on October 15 in Washington and met with General H. B. Sayler, Chief, Research and Development Service, Ordnance Department, and members of his staff. The personnel of the committee and information about the meeting of the committee appear in the December, 1947, issue of ASTM BULLETIN. The Ordnance Department has requested the Advisory Committee to advise it on research projects in three materials fields: (1) Plastics, covering two researches, namely, effect of petroleum base oils and fuels on plastics, and the aging of plastics; (2) adhesives, covering the factors leading to the deterioration of adhesive bonds; and (3) rubber, covering research into the fundamental mechanism of oxidation of rubber and other factors affecting the aging of rubber. For this purpose the Ordnance Advisory Committee has formed three small advisory panels, which under the procedure that has been established are available for direct conference on these matters with the appropriate personnel of the Ordnance Department.

The Society's advice respecting certain materials problems has also been sought by the Army-Navy Electronic and Electrical Standards Agency. This Agency prepared a statement on materials problems in the electrical insulation field which, after conference with the head of the Agency, Col. L. J. Tatom, and his staff, was referred to the two committees of the Society directly interested, namely, D-9 on Electrical Insulating Materials and D-20 on Plastics. Comments by these two committees on the Agency's statement of these problems have been submitted to the Agency with the assurance that through these committees the Society is prepared further to discuss these problems and researches on them with the Agency.

Periodic staff conferences with the Society of Automotive Engineers have been instituted to facilitate still closer collaboration between the two societies in the development of specifications covering materials used in the automotive field. Through cross-representation on the committees of the two societies there is already an excellent degree of collaboration, which will be facilitated by these staff conferences covering specifications in such fields as metals, petroleum products and lubricants, rubber, textiles, and plastics.

The Society has agreed to the dissolution of the American Coordinating Committee on Corrosion, of which it has been one of the members, and the transfer of the activities of that committee, including its assets, to the National Association of Corrosion Engineers, which has undertaken to carry on the objectives for which the Coordinating Committee was formed some ten years ago.

The Society has formed a Joint Committee on Soap Analysis with the American Oil Chemists Society, which functions under the Society's Committee D-12 on Soaps and Other Detergents.

The following appointments are announced: Prof. W. C. Voss as the Society's representative on the Division of Engineering and Industrial Research, National Research Council; Prof. K. B. Woods as the Society's representative on the Highway Research Board of the National Research Council; and R. W. Crum as the Society's representative on the American Documentation Institute.

American Standards Association:

The Board of Directors has had under consideration during the year several important administrative matters concerning the Society's membership in the American Standards Association. One of these is the matter of incorporation of ASA, concerning which there have been references in previous reports of the Board to the Society. An amendment of the Constitution of ASA has been adopted, on which the Board directed that an affirmative vote for A.S.T.M. be cast, which empowers the Board of Directors of ASA by a vote of not less than three-fourths of the member bodies to incorporate ASA as a membership corporation pursuant to the provisions of any applicable Federal statute or state law. Recently at a conference of the member bodies of ASA the member bodies voted in favor of immediate state incorporation of the Association with the understanding that the ASA Board of Directors should continue its efforts looking to Federal incorporation. In accordance with this action papers for state incorporation of the Association are being prepared and at the same time bills for Federal incorporation have been introduced in both the Senate and House of Representatives of the Congress. Certain provisions of these bills dealing with the matter of membership of Government agencies in ASA are presently under consideration in your Board.

The Board also has under consideration and is discussing with the Society's representatives on the Standards Council a proposal to amend the By-laws of ASA that would permit correlating committees of the Association to serve as sponsors for standardization projects.

The Board has followed with interest the work of a Special Survey Committee appointed by the Board of Directors of ASA which for the past few months has been engaged in examination of ASA operations, including a study of the organization of the ASA staff. Members of the committee visited the staffs of a number of the member bodies, in connection with which a worth-while conference with the staff of A.S.T.M. was held. The Survey Committee has prepared a statement of objectives and policies of ASA which was endorsed by the ASA Board and it is expected the member bodies will shortly receive copies of the report of the Survey Committee recently presented to the ASA Board.

There was an unusually large number of A.S.T.M. specifications and methods of test submitted to the ASA through several different procedures for approval as American Standard. The report of the Administrative Committee on Standards (Appendix II) gives further information on this phase of the Society's participation in ASA activities.

The Conference of Staff Executives of Member Bodies of ASA, of which the Executive Secretary is the chairman, held a meeting in February, 1948, at which a very interesting and informative discussion took place centered on two papers presented at the Conference: "What Standardization Can Mean to Industry—as Expressed Through Trade Associations," by A. S. Boisfontaine, Southern Pine Inspection Bureau, and "Trade Association Policies, Organization, and Procedure in the Development of Standards," by Charles M. Parker, American Iron and Steel Institute. The meetings of this Conference are serving to spread information about standardization activities in industry among not only the executives of member and associate member bodies of ASA but among officers of trade and other associations who are not in ASA.

H. H. Morgan has been re-elected for a term of three years beginning January, 1948, as a Director of ASA upon nomination of this Society. Miles N. Clair has been re-appointed for a term of two years beginning January, 1948, as the Society's representative on the Building Code Correlating Committee, and Myron Park Davis has been re-appointed for a term of two years beginning January, 1948, as the Society's representative on the Safety Code Correlating Committee. The Technical Secretary serves as alternate in the case of each of these appointments.

International Activities:

During the year the organization of the International Organization for Standards (ISO) has been effected through the appointment of a General Secretary, Henry St. Leger, and the establishment of permanent headquarters in Geneva. The second meeting of the ISO Council is being held there in June, 1948. During a visit last September to this country, Mr. St. Leger called at Society headquarters and discussed with the staff many matters of mutual interest, including the nature and extent of A.S.T.M. standardization activities and their international implications. We are keeping the ISO office currently informed of A.S.T.M. standardization work.

The International Electrotechnical Commission (IEC) has voted to affiliate with ISO under stipulated terms that are agreeable to each body and have been accepted by the U. S. National Committee of IEC. Our Society as a member of the latter committee voted in favor of such affiliation.

Suggestions made by the Board of Directors to ASA for changes in the Constitution and Rules of Procedure of ISO are still in the hands of a special committee of ASA for consideration.

Procedures for handling ISO standardization projects in this country, involving relationships between ASA and A.S.T.M. in the field of materials standards, are in course of development. Proposed projects presently under consideration include petroleum products, textiles, rubber, and steel.

With respect to petroleum products,

America held the secretariat for the petroleum standardization project under the former ISA, and consideration is now being given by ASA and A.S.T.M. to the possibility of continuing such secretariat under ISO through the offices of Sectional Committee Z11 on Petroleum Products and Lubricants, of which the Society is sponsor.

As to textiles, Sectional Committee L-14 on Textile Test Methods, of which the Society is joint sponsor with the American Association of Textile Chemists and Colorists, has proposed that the secretariat for the ISO project on Textile Materials, or at least that portion relating to methods of testing, be assigned to America. Prof. H. J. Ball, past-president of the Society, and chairman of Committee D-13 on Textile Materials, and A. G. Scroggie, vice-chairman of the committee, are attending a conference on this subject in England in June, 1948. They will also attend a British Congress on Textiles.

An inquiry as to whether America wishes to participate in an ISO project on Rubber is under consideration in Committee D-11 on Rubber and Rubber-like Materials. In the meantime H. G. Bimmerman, a member of the committee, will attend a meeting of ISO Committee 45 on Rubber to be held in London during June, 1948.

Finally, as to American participation in a possible ISO project on Iron and Steel, this question has been referred to Committee A-1 on Steel for its consideration and recommendations. Other projects under discussion are those on Viscosity, Shellac and Scientific Glassware.

The Society is participating in the International Conference on Soils Mechanics and Foundations, held in Delft, Holland, June, 1948, being officially represented at the Conference by Professors E. J. Kilcawley and F. J. Converse, respectively, chairman and vice-chairman of Committee D-18 on Soils for Engineering Purposes. A paper on the Society's work in the testing of soils will be presented at the Conference, and a report of the proceedings will be made to Committee D-18 and the Society.

Three committees of the Society have accepted invitations to be represented at the Rubber Technology Conference in London this June. Committees D-9 on Electrical Insulating Materials and D-20 on Plastics will be represented by A. J. Warner, and Committee D-11 on Rubber and Rubber-like Materials by H. G. Bimmerman.

Respectfully submitted on behalf of the Board of Directors,

T. A. BOYD,
President.

C. L. WARWICK,
Executive Secretary.

June, 1948.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1 TO DECEMBER 31, 1947

Philadelphia, January 8, 1948

Mr. C. L. Warwick, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the year ended December 31, 1947. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the records. Accounts receivable were not verified by correspondence with debtors. The investments owned by the Society were examined by us; all income from investments for the period under review was properly accounted for.

We have prepared and submit herewith balance sheet as of December 31, 1947, statement of cash receipts and disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of the Society.

In our opinion, these statements, with notes thereto, present fairly the financial position of the Society at December 31, 1947 and the results of its operations for the year ended that date.

Respectfully submitted,

John Heins & Co.
(Signed) I. RUSSELL BUSH
Certified Public Accountant

BALANCE SHEET AS OF DECEMBER 31, 1947

(Including Special and Designated Funds)

Assets

General Funds:			
Current: Cash	$42 430.42		
Less Check No. 3598 as of December 31, 1947 (estimated cost of 1947 Proceedings and 1947 Supplement to 1946 Book of Standards)	38 500.00		
		$3 930.42	
Investments (Market Value $157 372.94)—Cost		163 690.12	
Accounts Receivable		27 218.85	
Note Receivable Building Fund		10 000.00	
			$204 839.39
Furniture and Fixtures (depreciated book value)			16 405.39
Total Assets General Funds			$221 244.78
Building Fund:			
Cash		$6 274.97	
Investments (Market Value $15 042.18)—Cost		15 234.43	
Land (Cost)		30 000.00	
Building and construction costs	$148 865.26		
Less Reserve for depreciation	3 200.00	145 665.26	
Total Assets Building Fund			$197 174.66
Other Special and Designated Funds:			
Cash: A.S.T.M. Research Fund	4 144.92		
Dudley Medal and Marburg Lecture Fund	1 493.70		
Richard L. Templin Award Fund	51.96		
Committee Funds	15 080.51	20 771.09	

Investments:			
A.S.T.M. Research Fund (Market Value $40 689.64) Cost	43 251.36		
Dudley Medal and Marburg Lecture Fund (Market Value $4 043.18) Cost	6 625.00		
Richard L. Templin Award Fund (Market Value $1 002.81) Cost	1 000.00		
Sam Tour Award Fund (Market Value $1 482.00) Cost	1 500.00		
Committee Funds (Market Value $9 113.74) Cost	9 000.00	61 376.36	
Total Assets, Special and Designated Funds			$82 147.45
Total Assets			$500 566.89

LIABILITIES

General Funds:			
Current: Accounts Payable:			
Committee A-1	$489.68		
Committee C-1	170.12	$659.80	
Advance Collections			7 096.15
Total Current Liabilities—General Funds			7 755.95
Life Membership Fund	3 812.50		
Publication Fund	6 951.62		
Book of Standards Reserve	20 000.00		
Executive Retirement Reserve	10 380.00		
Retirement Fund Reserve	2 926.62		
Reserve for Depreciation of Investments	12 000.00		
Surplus	157 418.09	213 488.83	
Total Liabilities General Funds			$221 244.78
Building Fund:			
Contributions		163 732.43	
Accumulated Income and Profit on Investments		1 442.23	
Mortgage Payable, dated December 23, 1943, due within 5 years, with quarterly installments of $187.50 at 5% $23 875.00 reduced to		22 000.00	
Note Payable to A.S.T.M. General Funds		10 000.00	
Total Building Fund			$197 174.66
Other Special and Designated Funds:			
A.S.T.M. Research Fund:			
Principal	43 335.23		
Income	4 061.05	47 396.28	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	1 493.70	8 118.70	
Richard L. Templin Award Fund:			
Principal	1 000.00		
Income	51.96	1 051.96	
Sam Tour Award Fund:			
Principal		1 500.00	
Committee Funds, Unexpended Balances		24 080.51	
Total Special and Designated Funds			$82 147.45
Total Liabilities and Funds			$500 566.89

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1943–1947 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1943	$29 220.41	$111 136.68	$14 567.65	$6 960.38	$5 569.02	$17 113.98	$33 541.26	$105 660.86
1944	42 637.45	114 237.61	11 986.97	6 780.66	9 009.15	17 113.98	37 913.05	111 606.51
1945	46 058.60	153 718.05	15 033.90	6 641.57	5 432.16	15 000.00	35 982.49	165 037.47
1946	5 100.03	160 383.60	26 150.27	14 964.50	7 270.11	15 000.00	40 117.54	144 210.75
1947	3 930.42	163 690.12	37 218.85	16 405.39	7 755.95	20 000.00	36 070.74	157 418.09

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1947 TO DECEMBER 31, 1947

Cash balance, January 1, 1947			$5 100.03

RECEIPTS

Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$133 932.52		
Past Dues	992.00		
Advance Dues	3 936.00		
Income, Life Membership Fund	150.00		
Entrance Fees	5 420.00		
Total Dues		$144 430.52	
Sales of Publications:			
Book of Standards (Members, addl. parts)	33 646.38		
1944 Book of Standards and Supplements	2 966.37		
1946 Book of Standards	96 523.65		
1947 Supplements to Book of Standards	22.00		
Methods of Chemical Analysis of Metals	4 023.04		
Compilations of Standards	44 637.31		
Separate Standards	11 907.24		
Selected Standards for Students	8 388.82		
Proceedings	5 434.49		
Spring Meeting Papers	2 095.65		
Bulletin Subscriptions (non-members)	1 437.47		
Special and Miscellaneous Publications	40 626.01		
Total Sales of Publications		$251 708.43	
Miscellaneous:			
Advertising—Bulletin	14 943.27		
Advertising—Index	2 102.50		
Interest and Dividends	6 967.61		
Registration and Other Fees—Meetings	5 054.00		
Committee A-1 for Technical Assistant	2 482.05		
Miscellaneous	375.54		
Total Miscellaneous Items		$31 924.97	
Total Operating Receipts (budgeted)		$428 063.92	

Nonoperating Receipts (Not Budgeted):

Brought Forward	428 063.92		
Excess Remittances	798.84		
Committee C-1 for Technical Assistant	2 300.00		
Executive Retirement Reserve	300.00		
Retirement Fund Reserve	25.23		
Joint AIME-ASTM Publication Fund	872.50		
Annual Meeting Dinner	1 355.00		
Total Nonoperating Receipts		$5 651.57	
Total Receipts			$433 715.49
Total Receipts for 1947 and Cash Balance January 1, 1947			$438 815.52

DISBURSEMENTS

Operating Disbursements (Budgeted):

Publications:			
Year Book	$7 048.38		
1946 Book of Standards	27 145.24		
1947 Supplement to Book of Standards	22 302.70[a]		
Compilation of Standards	16 212.23		
Separate Standards	4 252.44		
Selected Standards for Students	5 099.77		
Chemical Analysis of Metals	48.45		
Proceedings	27 979.64[a]		
Preprints	4 803.44		
Spring Meeting Papers	2 717.88		
A.S.T.M. Bulletin	18 728.40		
Circulars to Members	3 421.90		
Index to Standards	6 779.49		
Special and Miscellaneous Publications	29 076.99		
Total Disbursements—Publications		$175 616.95	
Salaries		144 600.57	
General Office Expenses		33 925.39	
Expenses—Technical and District Committees		11 685.70	
Expenses—Meetings		9 858.82	
American Standards Association		1 000.00	
Traveling Expenses, Administrative and Special Committees		4 383.25	
Headquarters Occupancy Expenses (includes $3200 depreciation on building)		14 013.38	
Furniture and Fixtures		3 677.99	
Dues, Contributions, Miscellaneous		412.90	
Legal Services		1 400.00	
Employees Retirement Fund		9 321.57	
Real Estate Agent's Fee		500.00	
Total Operating Disbursement (Budgeted)		$410 396.52	

Nonoperating Disbursements (Not Budgeted):

Refund of Excess Remittances	798.84		
Investments	3 306.52		
Technical Assistant Committee C-1	2 205.87		
Joint AIME-ASTM Publication Fund	161.25		
Book of Standards Reserve	15 000.00		
Special Award Fund	1 500.00		
Annual Meeting Dinner	1 516.10		
Total Nonoperating Disbursements		$24 488.58	
Total Disbursements			$434 885.10
Cash Balance, December 31, 1947			$3 930.42

---

[a] These accounts include $38 500.00 representing a check drawn against cost of the 1947 Proceedings and 1947 Supplement to 1946 Book of Standards, but not actually paid on December 31, 1947.

A.S.T.M. BUILDING FUND

DETAILS OF BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JANUARY 1, 1947 TO DECEMBER 31, 1947

Cash Balance, January 1, 1947		$3 908.47
Receipts		
Contributions 1947	$9 312.00	
Income from Investments:		
Interest on U. S. Treasury Bonds 2½s 1967–72	469.78	
Proceeds from sale of $10 000 U. S. Treasury Bonds 2½s 1967–72	10 321.88	
From General Funds for Depreciation on Building and Elevator	3 200.00	23 303.66
		$27 212.13
Disbursements		
Architect's Fees	$1 936.37	
General Contractor	7 519.85	
Air Conditioning Contract	10 096.06	
Mat for Main Entrance	34.88	
Real Estate Agent's Fee	500.00	
Interest on Loan from General Funds	100.00	
Reduction of Mortgage—4 quarterly payments of $187.50 each	750.00	20 937.16
Cash Balance, December 31, 1947		$6 274.97

A.S.T.M. BUILDING FUND

Details of Assets and Liabilities as of December 31, 1947

Assets

Cash in Girard Trust Co., Philadelphia, Pa.				$6 274.97
Investments:				
$15 000. U. S. Treasury Bonds 2½s 1967–72 at $101.16 (Market Value $15 042.18)—Cost				15 234.43
Property, 1916–18 Race Street, Philadelphia, Pa.:				
Purchase Price	$53 625.00			
Commission to Agent	2 681.25			
Settlement fees, net	197.10			
	$56 503.35			
Allocated to:				
Land			$30 000.00	
Building		$26 503.35		
Construction Costs:				
Architect's fee	10 636.37			
Rent paid to facilitate possession	110.00			
Zoning fees and permits	17.50			
General Contractor	86 365.72			
Air Conditioning	17 310.72			
Elevator	7 548.04			
Air Conditioning Unit	210.00			
Real Estate Agent's Fee	500.00			
Miscellaneous	816.48			
	123 514.83			
Less, Proceeds from sales and salvage of equipment and rentals	1 152.92	122 361.91		
		148 865.26		
Reserve for Depreciation (provided from General Funds)		3 200.00	145 665.26	175 665.26
Total Assets				$197 174.66

Liabilities and Funds

Contributions		$163 732.43	
Income on Investments:			
Interest on U. S. Treasury 2½s 1967–72	$1 376.55		
Less Interest on Loan from General Funds	100.00	1 276.55	
Profit on Sale of $10 000. U. S. Treasury Bonds		165.68	
Mortgage Payable—dated December 23, 1943, due within 5 years with quarterly installments of $187.50 each, at 5 per cent, assumed 1945	23 875.00		
Less, 10 quarterly payments of $187.50 each	1 875.00	22 000.00	
Note Payable to A.S.T.M. General Funds		10 000.00	$197 174.66

In addition to the General Funds of the Society the Treasurer had on hand the following funds as of December 31, 1947:

A.S.T.M. RESEARCH FUND

	Total	*Invested*	*Uninvested Cash*
Principal Account:			
Balance, January 1, 1947	$43 335.23	$41 082.03	$2 253.20
Receipts: none			
Disbursements:			
Purchase of Securities		+2 169.33	−2 169.33
Balance, December 31, 1947	$43 335.23	$43 251.36	$83.87
Income Account:			
Balance, January 1, 1947		$4 361.76	
Receipts:			
Interest and Dividends on Investments		1 549.29	$5 911.05
Disbursements:			
Administrative Committee on Ultimate Consumer Goods		600.00	
Advisory Committee on Corrosion		500.00	
Engineering, Foundation, Welding Research		250.00	
University of Illinois, Committee A-3, Tensile and Fatigue Tests of Cast Iron		500.00	1 850.00
Balance, December 31, 1947			$4 061.05

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1947			
Principal—Investments (at cost)	$6 625.00		
Income—Cash	253.18	$6 878.18	
Receipts			
Interest on Investments		1 446.52	$8 324.70
Disbursements			
Honorarium		200.00	
Lecture Certificate		6.00	206.00
			$8 118.70
Accounted for as follows:			
Principal—Investments (at cost)		6 625.00	
Income—Cash in bank		1 493.70	$8 118.70

RICHARD L. TEMPLIN AWARD FUND

Balance, January 1, 1947			
Principal—Investments (at cost)	$1 000.00		
Income—Cash	113.46	$1 113.46	
Receipts			
Interest on Investments	25.00		
Contribution from General Funds	50.00	75.00	$1 188.46
Disbursements			
Templin Award		25.00	
Cost of Certificate Plate		111.50	136.50
			$1 051.96
Accounted for as follows:			
Principal—Investments (at cost)		$1 000.00	
Income—Cash in bank		51.96	$1 051.96

SAM TOUR AWARD FUND

Principal—Investments (at cost)	$1 500.00

COMMITTEE FUNDS

Committee A-1 on Steel	$744.82	
Committee A-5 on Corrosion of Iron and Steel	2 542.98	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	2 661.44	
Committee B-6 on Die-Cast Metals and Alloys	2 599.55	
Committee B-8 on Electrodeposited Metallic Coatings	11.25	
Committee C-1 on Cement	818.26	
Committee C-1 Master Builders Co.—Special Fund	863.23	
Committee C-1 Cement Reference Laboratory	538.56	
Committee C-9 Sanford E Thompson Medal Fund	964.77	
Committee C-9 on Concrete and Concrete Aggregates	153.32	
Committee C-15 on Manufactured Masonry Units	378.56	
Committee C-18 on Natural Building Stones and Slate	176.36	
Committee D-1 on Paint, Varnish, Lacquer and Related Products	315.05	
Committee D-2 on Petroleum Products and Lubricants	57.57	
Committee D-2 D.C.C. Reference Fuel Account	4 000.00	
Committee D-4 on Road and Paving Materials	36.95	
Committee D-5 on Coal and Coke	1 445.24	
Committee D-12 on Soaps and Other Detergents	19.08	
Committee D-13 on Textile Materials	894.08	
Committee D-13 on Research Textile Finishes	238.34	
Committee E-3 on Chemical Analysis of Metals	55.11	
Committee E-9 on Fatigue	197.56	
A.S.A. Sectional Committee on Specifications for Cast Iron Pipe and Special Castings	1 661.95	
Detroit District Council	1 183.33	
Philadelphia District Council	405.96	
Pittsburgh District Council	55.50	
Western New York—Ontario District Council	360.14	
Advisory Committee on Corrosion	500.00	
Administrative Committee on Ultimate Consumer Goods	201.55	
Total Committee Funds		$24 080.51
Accounted for as follows:		
Cash in banks	$15 080.51	
Investments (at cost)	9 000.00	
		$24 080.51

INVESTMENTS

AS OF DECEMBER 31, 1947

GENERAL FUNDS

Par Value		Cost or Book Value	Market Value[a]
	Bonds		
$2 000	Associated Electric Co. 4½, 1953	$1 693.50	$2 010.00
4 000	Erie R. R. Co. Gen Mtg. Income 4½s, 2015	2 832.00	2 610.00
2 000	Hudson Coal Co. S. F. 5s Series A, 1962	1 697.25	1 750.00
3 000	Southern Pacific Deb. 4½s, 1969	2 544.75	2 700.00
13 000	U. S. Savings Bonds, Series G 2½s[b]	13 000.00	12 352.00
4 800	U. S. Treasury Bonds, 2½s, 1967–72	4 800.00	4 813.50
2 000	U. S. Treasury Bonds, 2½s, 1964–69	2 000.00	2 015.63
2 000	U. S. Treasury Bonds, 2s, 1952–54	2 000.00	2 023.13
50 000	U. S. Treasury Bonds, 2s, 1952–54	50 765.63	50 578.13
5 000	Walworth Co. 3¼s convertible debentures, 1976	5 496.53	4 431.25
		$86 829.66	$85 283.64
Shares	*Preferred Stocks*		
22	American Airlines Inc., 3½ cumulative convertible, preferred	2 244.00	1 149.50
19	American Cyanamid Co., 5 per cent cumulative preferred	190.00	200.29
10	American Cyanamid Co., 3½ per cent Series A convertible preferred	1 042.53	1 050.00
25	Colgate-Palmolive-Peet Co., $3.50 preferred	2 525.00	2 175.00
100	Murray Corp. of America, cumulative preferred 4 per cent	5 050.06	4 350.00
20	Philco Corp., 3¾ per cent Series A preferred	2 030.00	1 710.00
		$13 081.59	$10 634.79
	Common Stocks		
75	Allis-Chalmers Mfg. Co., common, no par value	3 316.20	2 925.00
25	American Can Co., common	2 122.30	2 003.13
50	American Cyanamid Co., Class B. common	1 764.25	2 000.00
100	American Rad. & Std. Sanitary Corp., common	1 564.32	1 450.00
25	American Tobacco Co., common	2 110.12	1 675.00
30	American Viscose Corp., common	2 323.58	1 800.00
100	Atlantic Refining Co., common	2 302.50	3 875.00
100	Baldwin Locomotive Works, common	3 822.00	1 425.00
100	Carpenter Steel Co., common	5 426.00	4 000.00
10	Delaware Power and Light Co., common	135.00	167.50
75	General Telephone Corp., common	2 675.00	2 015.63
100	International Harvester, common	9 610.00	8 900.00
60	Lone Star Cement, common, no par value	4 349.42	4 020.00
100	S. S. Kresge Co., common	2 490.18	3 850.00
100	Mathieson Alkali Works, common	3 016.00	3 275.00
30	Montgomery Ward Co., common	1 813.99	1 601.25
75	J. C. Penney Co., common	2 279.63	3 225.00
67	Philadelphia Electric Co., common	1 185.06	1 574.50
116	Public Service Corp. of N. J., common	4 369.50	2 392.50
60	The Texas Company	2 969.55	3 607.50
20	United Gas Improvement Co., common	315.00	422.50
50	U. S. Gypsum, common	3 819.27	5 250.00
		$63 778 87	$61 454.51
	TOTALS	163 690.12	157 372.94

A.S.T.M. BUILDING FUND

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
$15 000	U. S. Treasury, 2½s, 1967–72	$15 234.43	$15 042.18

A.S.T.M. RESEARCH FUND

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
$2 000	Associated Electric Co. 4½s, 1953	1 693.50	2 010.00
1 500	Balto. & Ohio R. R. Gen. & Ref. 5s Series M. 1996	1 511.25	922.50
2 000	Hudson Coal Co. S.F. 5s Series A, 1962	1 697.25	1 750.00
4 500	U. S. Defense Savings Bonds, Series G. 2½s[b]	4 500.00	4 276.50
2 000	U. S. Treasury Bonds 1965–70, 2½s	2 000.00	2 015.63
		$11 402.00	$10 974.63

Shares	*Preferred Stocks*		
30	Allis-Chalmers Mfg. Co. 3¼ cumulative convertible preferred	3 067.22	2 775.00
33	American Airlines Inc. 3½ cumulative convertible preferred	3 366.00	1 724.25
30	Butler Bros., Cum preferred	3 090.00	2 430.00
12	Consolidated Edison Co. of N. Y. $5 preferred	1 234.39	1 260.00
29	Consumers Power Co., $4.50 preferred, no par value	2 914.50	2 903.63
20	Philco Corp., 3¾ Series A preferred	2 030.00	1 710.00
		$15 642.11	$12 802.88

	Common Stocks		
100	Allis-Chalmers Mfg. Co., common, no par value	4 421.59	3 900.00
5	Delaware Power & Light Co., common	67.50	83.75
98	General Motors Corp. common, par $10—gift[c]	6 504.75	5 708.50
20	Montgomery Ward Co., common	1 209.33	1 067.50
100	Ohio Oil Co., common	2 167.88	3 300.00
33	Philadelphia Electric Co., common	583.70	775.50
9	Public Service Corp. of N. J., common	135.00	185.63
10	United Gas Improvement Co., common	157.50	211.25
16	U. S. Gypsum Co., common	960.00	1 680.00
		$16 207.25	$16 912.13
	TOTALS	43 251.36	40 689.64

---

[a] Market values were taken from current financial publications.
[b] Market Value is stated at redemption value as of December 31, 1947.
[c] The income from these shares is, by agreement, paid to an annuitant.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Par Value	*Bonds*	*Cost or Book Value*	*Market[a] Value*
$500.00	Balto. & Ohio R. R. Ref. & Gen. Mtg. 6s, Series J., 1995	$550.00	$353.18
6 000.00	Balto. & Ohio R. R. Ref. & Gen. Mtg. 5s, Series G., 1995	6 075.00	3 690.00
		$6 625.00	$4 043.18

RICHARD L. TEMPLIN AWARD FUND

$1 000.00	U. S. Treasury Bonds $2\frac{1}{2}$s, 1967–72	$1 000.00	$1 002.81

SAM TOUR AWARD FUND

$1 500.00	U. S. Defense Savings Bonds Series G, $2\frac{1}{2}$[b]	$1 500.00	$1 482.00

COMMITTEE FUNDS

$1 000.00	U. S. Treasury Bonds, $2\frac{7}{8}$s, 1960–55	$1 000.00	$1 066.25
2 000.00	U. S. Treasury Bonds, $2\frac{1}{2}$s, 1964–69	2 000.00	2 015.63
3 000.00	U. S. Treasury Bonds, $2\frac{1}{2}$s, 1965–70	3 000.00	3 023.43
3 000.00	U. S. Treasury Bonds, $2\frac{1}{2}$s, 1967–72	3 000.00	3 008.43
		$9 000.00	$9 113.74

GRAND TOTAL, Common stocks	$79 986.12	$78 266.64
GRAND TOTAL, Preferred stocks	28 723.70	$23 437.67
GRAND TOTAL, Bonds	131 591.09	126 942.18
	$240 300.91	$228 746.49

---

[a] Market values were taken from current financial publications.
[b] Market Value is stated at redemption value as of December 31, 1947.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

While the Committee on Standards has a very broad function in connection with all of the Society's standardization activities, much of the committee's attention during the past year has been devoted to routine matters such as reviewing and taking action on recommendations submitted to it by the technical committees concerning proposed tentatives, tentative revisions of standards, and revisions of tentatives. The number of such recommendations coming to the committee continues to be large. Only one meeting was held, on September 4, 1947, at which such recommendations were considered. However, quite a number of recommendations were also acted upon by letter. As a result of actions taken, 32 new tentatives were accepted as well as 41 revisions of tentatives, 7 tentative revisions, 10 standards reverted to tentative, and 3 tentatives withdrawn. Usually the letter ballot vote in the technical committees on recommendations is quite clear with a preponderant affirmative vote recorded. Even in such cases, however, negative viewpoints are carefully scrutinized so that the Administrative Committee can feel assured that they have been properly considered in the technical committee concerned. One or two instances have come before the committee where a fair number of negative votes had been recorded. These cases were considered at a meeting of the Standards Committee and all viewpoints presented and discussed.

As a matter of record a list of the recommendations approved appears as an appendix to the report.[1]

Apart from actions on standards, the committee has had before it proposals for new projects that should be undertaken, and in addition is continuing to give considerable attention to the character and quality of the Society's standards in general.

Expansion of Standardization Activities:

Of the projects referred to in the report presented a year ago, organization has been completed of Committee C-19 on Structural Sandwich Constructions with the holding of an organization meeting on January 30, 1948.

Appearance.—A Committee on Appearance has been authorized by the Board, and the personnel is now being assembled in cooperation with the various technical committees of the Society that are interested in the subject. To a large extent this committee will function as a coordinating agency and the committee is being given the designation of E-12.

Acoustical Materials—In the report of a year ago it was recommended that a conference be held of those interested to consider the desirability of organizing a committee on acoustical materials. Such a conference has been held, and on the basis of its recommendations the Board of Directors has a definite proposal that a committee on acoustical materials be organized. In connection with such a committee, acoustics is considered to relate to airborne sound. Certain types of materials sometimes referred to as acoustical materials but whose function is that of vibration absorption or damping are considered to be outside the scope of the proposed committee.

---

[1] A list of recommendations approved by the Standards Committee was appended to the report as submitted to the Board of Directors. It is not reproduced here since the recommendations have all been announced currently throughout the year in the various issues of the ASTM BULLETIN.

White Wares.—The attention of the committee has recently been called to a proposal by the American Ceramic Society to having the standardization work in this field handled in an A.S.T.M. committee in a manner similar to that in which the A.C.S. looks to A.S.T.M. Committee C-8 for work on refractories and A.S.T.M. Committee C-14 for standardization work in the field of glass. An official request from the A.C.S. looking toward the organization of a new A.S.T.M. committee is now before the Board of Directors for consideration. The scope of this committee would include sanitary ware, electrical porcelain, laboratory ware, and dinner ware.

Porcelain Enamel.—A subject closely allied to that above is that of porcelain or ceramic enamels. The committee is now making a canvass as to the desirability of organizing an A.S.T.M. committee in this field. It too would cover certain types of sanitary ware but would also include finishes applied to a number of articles, as for example refrigerators, and certain types of construction materials.

Quality and Form of Standards:

Pursuant to the 1947 report of this committee, the Chairman wrote to the various technical committee chairmen, bringing to their attention the desirability of obtaining the highest quality in A.S.T.M. standards from both editorial and technical points of view. The letter furthermore suggested the establishment of special editorial subcommittees within the technical committees for the purpose of reviewing proposed standards as an aid in obtaining the desired quality. As of this date, the following action has been taken by the technical committees: Eleven committees already had editorial or similar subcommittees charged with reviewing proposals prior to Standards Committee action; fifteen additional committees have established such subcommittees; fourteen committees have decided that their situation does not require such action; twenty-one committees have either not considered the matter or have not furnished information regarding action taken.

While we believe that some improvement has taken place during the past year in the quality of standards actions proposed to the Society, it is proposed to follow the matter further with a number of the technical committees with the aim of effecting a further improvement during the coming year.

Relations with American Standards Association:

Two methods under the procedure of the American Standards Association are used by A.S.T.M. in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing A.S.T.M. standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the A.S.T.M. is granted proprietary sponsorship so far as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, but with A.S.T.M. as sponsor or cosponsor for the sectional committee.

Standards Submitted Under the Existing Standards Procedure.—A number of new standards have been submitted during the past year under the existing standards procedure. These consist of a group of 20 specifications for soaps and other detergents, and the Standard Methods of Sampling and Chemical Analysis of Special Detergents (D 501–46), all under the jurisdiction of A.S.T.M. Committee D-12 on Soaps and Other Detergents. These have been submitted for approval as American Standard at the request of the ASA Consumer Goods Committee since many of them cover materials of direct interest to the ultimate consumer.

Revised Standards Submitted Under Proprietary Procedure.—In view of the Society having been designated as proprietary sponsor for the revisions of A.S.T.M. standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 12 specifications relating to steel, 2 relating to malleable iron, 2 relating to zinc coatings on structural steel, 3 relating to copper and copper alloys, and 8 relating to pigments.

Standards Submitted Under Sectional Committee Procedure.—During the year reports were received from 5 of the sectional committees for which the Society is sponsor or joint sponsor as follows:

Sectional Committee A1 on Portland Cement.—This committee has recommended for approval as American Standard 1 tentative specification, and 13 methods.

The committee had also intended to recommend approval of the A.S.T.M. Standard Specifications for Portland Cement (C 150 – 46) and A.S.T.M. Specifications for Air-Entraining Portland Cement (C 175 – 46 T), but an objection had been raised to a requirement which was not in accordance with the requirement of the specifications of the American Association of State Highway Officials, and accordingly this recommendation was temporarily withheld.

Sectional Committee A37 on Road and Paving Materials.—This committee has recommended for approval as American Standard 14 A.S.T.M. specifications, 27 A.S.T.M. methods, a group of A.S.T.M. definitions of terms relating to materials for roads and pavements, 1 A.S.T.M. recommended practice for bituminous paving plant inspection, and a volume correction table for tar and coal-tar pitch, together with 2 specifications and 7 methods of the AASHO.

The committee has also recommended the reaffirmation of 13 standards.

It is of interest to note that this sectional committee has been instrumental in reconciling differences in a number of standards of the A.S.T.M. and AASHO.

Sectional Committee C59 on Electrical Insulating Materials.—This committee is recommending the approval of 4 revised American Standards.

Sectional Committee L14 on Textile Test Methods.—This committee is recommending for approval as American Standard 37 A.S.T.M. methods, and 11 methods of the American Association of Textile Chemists and Colorists.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee is recommending the reapproval as American Standard of 4 revised A.S.T.M. standards, and the withdrawal as American Standard of 2 A.S.T.M. methods.

All of these reports have been referred to the American Standards Association and action is pending for the most part on the recommendations concerned.

International Relations:

There has been renewed activity with respect to the relations with standardizing bodies of other countries. A number of them, principally in the British Commonwealth, are referring draft standards to the A.S.T.M. for comment, and some have put the A.S.T.M. committees on their mailing list to receive copies of minutes of committee meetings.

The outstanding developments of the year, however, have related to the setting-up of the International Organization for Standardization (ISO) to take the place of the earlier International Standards Association (ISA) and the interim United Nations Standards Coordinating Committee. The preliminary steps in organizing the ISO were discussed in the report a year ago and reference made to some few topics that were under consideration. During the year the Society has been approached with respect to several ISO projects that are now being set up as indicated below:

ISO Project on Petroleum Products is a continuation of the old ISA Committee 28 for which America held the secretariat, and Sectional Committee Z11 on Petroleum Products and Lubricants, for which the Society is sponsor, is giving consideration to the manner in which the secretariat might be handled for America. This sectional committee will also be looked to for recommendations as to American representation on the ISO committee.

The organization meeting of the ISO Committee on Rubber is being held in London during June, 1948. Mr. H. G. Bimmerman, E. I. du Pont de Nemours and Co., on the recommendation of A.S.T.M. Committee D-11 on Rubber and Rubber-like Materials, is the American representative.

The organization meeting of the ISO Committee on Textiles is being held in Buxton, England during June, 1948. Since this is a project of very broad interest to American industry, a rather extensive delegation is going abroad to attend the organization conference. Among them are H. J. Ball, Chairman of A.S.T.M. Committee D-13, on Textile Materials, and A. G. Scroggie, Vice-Chairman of Committee D-13, who have been nominated by Committee D-13.

There was referred to the Society for its recommendation the proposal that an international project be organized on determination of viscosity. This matter was referred to Committee E-1 on methods of testing, which committee recommended the establishment of such an international project but felt that the work should be confined to the establishment of reference standards and standardization of apparatus and procedures for measuring absolute viscosity. Committee E-1 further recommended that America should accept the secretariat for the project.

In addition, the advice of the Society has been asked regarding the establishment of a project on shellac under the auspices of the Indian Standards Institution; on scientific glassware under the auspices of great Britain; and on iron and steel, also under the auspices of Great Britain. America was not represented in the earlier ISA committee on this latter subject.

Respectfully submitted on behalf of the committee,

R. D. BONNEY,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

Accomplishments:

The Administrative Committee on Research has held two meetings at Society Headquarters: the first a two-day meeting on October 16 and 17, 1947, and the second on April 16, 1948.

The meeting on October 16 was devoted primarily to a meeting with four A.S.T.M. technical committees as follows: Committee A-7 on Malleable Iron Castings; Committee B-9 on Metal Powders and Metal Powder Products; Committee D-6 on Paper and Paper Products; and Committee D-13 on Textile Materials.

These meetings with the various technical committees are a continuation of an activity started last year. The major objective is to find out in what way the Administrative Committee can be most useful in fulfilling its functions and particularly, in increasing research activities. The series of meetings has now established a fairly clear picture of the activities of technical committees; the methods of handling research; the amount and type of research in progress at the present time and contemplated for the future. The amount of research has an extremely wide range, all the way from no research in progress and no research contemplated to extensive research campaigns which have been worked up, put into operation by the committees, and adequately financed by them. As a result of these meetings, the Administrative Committee has been able to draw up organization charts which represent the functioning of various committees ranging from those with no research to those with comprehensive research programs.

While it seems probable that these meetings with the technical committees have most benefited the Administrative Committee, there is evidence that some benefits have been obtained by the committees which have attended these meetings. This is indicated by an increased interest in research planning, establishment of research subcommittees, and plans of two of the committees to undertake research work, which had not been in operation prior to these meetings.

It has developed as a result of meeting with technical committees, that there is a wide spread in the activities of the different committees in making adequate contacts with other committees working in related fields and with research laboratories and other organizations from which they might obtain valuable data for their committee activities. However, in the case of some committees, contacts have been made so that they have the benefit of researches involving large sums of money and carried out in the leading laboratories and organizations in their field. This is considered to be an important activity for all of the technical committees, and a method of improving the work of the committees which can be used to a greater extent with no extra cost to the committees or to the Society.

During the current year, the committee has made grants from the Research Income Fund to Committee A-3 on Cast Iron in support of the research at the University of Illinois on tension and fatigue tests on cast iron at elevated temperatures,[1] and to the Engineering Foundation in support of the activities of the Welding Research Council.

It has become evident that the Income Fund is so limited that its use must be restricted to catalyzing researches rather than supporting them. The grant to Committee A-3 seems to have functioned in this way because the committee has now authorized its Subcommittee XXII on Elevated Temperature Properties of Cast Iron to draw up plans for a comprehensive study of this subject.

Current Problems and Future Work:

The main problem before the Administrative Committee remains the finding of the best ways and means of accomplishing the objectives for which the committee was established. The major effort has been directed to promoting research by the Society, particularly by the various technical committees. A good sampling has been made of the committees so that it is now known to what extents they are engaging in research and the mechanism by which the various committees are operating. A study has been made to reduce the operations of the committees to simplified flow sheets or organization charts and two organization charts, one for small, and the other for large committees have been reviewed. As a result of this study, the Administrative Committee will shortly submit to the technical committees suggestions for stimulating their research activities.

Since it is impossible to finance, in a significant way, researches from the Income Fund, the committee has plans under way to encourage research by the committees, as indicated above; and further by supplying lists of research problems which are considered important in their field. It is proposed to have these problems originate at least in part, and probably for the most part, in the various technical committees. The research problems originating in the technical committees would be supplemented by suggestions from members of the Administrative Committee and be correlated with researches proposed by other committees. It would then be the plan to publish a list of researches which should be undertaken. The publication would be distributed not only to members of the Society but to educational institutions and various research laboratories which might be on the lookout for good research problems. It is thought that the funds available from the Research Income Fund may be adequate for the preparation and distribution of a report on research projects.

Special Recommendations:

It is believed that research by the technical committees should be encouraged. Ways and means of completing this objective have been discussed earlier in this report. Further efforts should be made along the line of cooperation between committees and by cross-representation on committees. Committees in many cases are not making full use of contacts with organizations which are doing extensive research in their field of activities or in related fields. In the case of some committees, excellent use is being made of such contacts and it is believed that other committees could improve their work by similar means, and it may be recommended that committees which have not made adequate contact with researches in their field should attempt to do so.

---

[1] This investigation was reported upon in a paper by W. L. Collins on "Fatigue and Static Load Tests of an Austenitic Cast Iron at Elevated Temperatures" which was presented at this annual meeting of the Society, see p. 696.

Consideration has been given by the Administrative Committee to the establishment of a substantial research endowment, income from which would make it possible to aid in a more material way in promoting research effort. Plans for securing such endowment funds are being considered, but as yet are only in the development stage. Special gifts, for which there is already a precedent, appear to be the most promising way for building up such an endowment fund. This problem is one for future consideration in the Administrative Committee on Research, in the Board of Directors and in the Society as a whole.

Respectfully submitted on behalf of the committee,

OSCAR E. HARDER,
Chairman.

C. L. WARWICK,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The Report of the Administrative Committee on Papers and Publications a year ago mentioned that an extensive study was being made of the publication setup of the Society. In making this study, two special committees had been appointed, one to examine the question as to how the standards might best be published, and the second to review the manner of publishing the technical papers. This latter, of course, involves the *Proceedings*, special technical publications, and the ASTM BULLETIN. Based upon the work of these two special committees, the Administrative Committee has submitted recommendations to the Board, as outlined in greater detail below. Briefly, these recommendations are largely to the effect that no major change be made in the present publication setup, such as the discarding of the annual *Proceedings* for a technical journal or the abandonment of our present Book of Standards for some other form of publication. It is proposed, however, that the number of issues of the ASTM BULLETIN be increased from six to eight with the continuation of the present type of material that is now being published therein; also that the Book of Standards be assembled in such a way as to make use of the compilations of standards in in specific fields, of which there are now 22. Certain economies could be effected by this proposal. Various suggestions for issuing the standards in loose leaf or loose unit form were carefully considered but were found to be inappropriate for A.S.T.M. use in view of the increased expense that would be entailed. Further discussion of the several recommendations of the committee are set forth below.

Two meetings of the committee were held during the year: a two-day meeting held November 17 and 18 and a meeting February 16. In addition several separate meetings were held of the special study committees.

PUBLICATION OF PAPERS AND REPORTS; ASTM BULLETIN

In undertaking its assignment to study the manner of publishing technical papers, the committee set up a small subcommittee of three of its members—P. G. McVetty (Chairman), G. R. Gohn, and K. B. Woods, who examined critically not only the Society's methods of publishing technical papers and reports but also the methods being followed by a number of other technical societies whose problems are more or less similar to ours. In the course of its work the subcommittee assembled much information having direct bearing upon its assignment, and providing essential background material.[1]

Recommendations:

The committe believes that no extensive changes in the Society's methods of publishing its technical papers and committee reports are necessary. It does not recommend the establishment of a monthly technical journal as the medium for publication of technical papers, but does recommend continued and expanded use of the ASTM BULLETIN for the publication of papers of current and general interest. The committee's study of the problem has led it to the conclusion that the Society's present publication structure is an excellent compromise of all the conflicting factors involved, and the committee has observed with some interest that several of our sister societies have recently made modifications in their publication policies that bring their methods closer to ours, and that these changes were made especially to bring about more expeditious publication of papers.

---

[1] This material should be of interest in studying the recommendations submitted herewith, and copies will be made available on request.

The committee's specific recommendations are:

As to Technical Papers

1. Continue present practice of preprinting as many papers received as possible and in addition printing separates of other papers as soon after the meeting as possible. Provide preprints for each session at the annual meeting.

2. Continue to publish papers of current and general interest in the BULLETIN.

3. Continue the present practice with bound volume of *Proceedings* at end of each year. A limitation might be placed on the size of the *Proceedings* of, say, 1200 pages of technical papers, with perhaps a closer screening so that only papers of definitely high quality would be accepted. Every means should be taken to improve the quality of the papers that are intended for publication in the *Proceedings*.

4. Continue publication of symposiums and papers in specialized fields as Special Technical Publications.

As to Committee Reports

1. Continue present plan of preprinting committee reports, and condense the reports to include only material of permanent reference value.

2. Publish progress reports in the BULLETIN.

3. Consider change from action on standards at annual meeting only to more frequent action by Committee on Standards.

4. Continue the present plan of revising committee reports after presentation but publish these as a separate paper bound volume, separate from technical papers, possibly distributed only on request.

As to ASTM BULLETIN

1. Publish eight issues of the BULLETIN a year instead of six.

2. Separate the technical articles from news matter to facilitate filing.

3. Number BULLETIN pages devoted to technical papers consecutively throughout the year.

4. For convenience, locate abstracts, if any, close to the table of contents.

5. Consider the establishment of an Editorial Board for the BULLETIN and the extension of editorials on subjects of current and general interest.

6. Consider articles on correlation of A.S.T.M. methods.

7. Increase technical articles to approximately eight per issue, giving preference to those of current and general interest.

PUBLICATION OF BOOK OF A.S.T.M. STANDARDS

With the continued and rapid growth in the number of standards and tentatives, there is serious question how much longer the present methods of publication will be either physically possible, or desirable. The 1946 edition of the Book of Standards is issued in five parts; in accordance with the present plans there will be no further complete reprinting for a three-year interval, and with supplements issued in 1947 and 1948 for each of the parts, at the end of this period 16 volumes would be involved, including the Methods of Chemical Analysis of Metals. Various suggestions that have been made from time to time for modification of the present scheme of publication have been considered, including publication in "loose-leaf" or "loose-unit" form, or publication in the form of a number of separate books covering the various groups of materials —much along the lines of the present "compilations" that have proved so useful and popular. The committee is also interested in ways and means of bringing the standards out earlier in the year and also reducing publication costs.

For this study, the committee formed another small subcommittee, comprising J. C. Geniesse (chairman) and G. R. Gohn. (A third member, W. H. Gardner, has since retired from the committee.) Their first study was on the proposal that a loose-leaf or loose-unit system be adopted in place of the bound volumes now used. Such a scheme would appear to entail less printing and thus result in a saving of paper and time, on the score that it would be necessary to print only the revised and new material each year, thereby eliminating the preparation of the complete Book of Standards every two or three years which includes a certain amount of standing material. Each owner of the Book of Standards would simply remove the out-of-date standards and insert the new ones. The plan has worked successfully in the case of some organizations.

A detailed study, which included assembling a considerable amount of comparative cost data, predicated on a six-year period (which should be the maximum any given binder could be used), showed, however, that in the case of A.S.T.M. such a plan would actually result in increased printing and handling costs. This is due primarily to the relatively large turnover in A.S.T.M. standards. The new or replacement material averages about one-third of the book each year. Added to this is the fact that a number of complete new books must be made available each year for new members and for sale, which means a relatively large advance accumulation of loose sheets or parts. Changes in any of this material would involve the waste of throwing away thousands of pages. The several points developed are itemized below:

1. There would be an increase in cost of $20,000 to $30,000 per year due to (*a*) extra labor for gathering and drilling and (*b*) the larger number of pages involved due to the number of blank pages entailed in loose-leaf or loose-unit publication.

2. The cost of ring binders would be an additional $120,000 over a six-year period.

3. In addition would be the extra cost of making up complete books from the original standards books and the supplements during the second to sixth years, which would be a relatively expensive hand operation.

4. Special tables of contents would need to be issued with every revision.

The above items do not include the additional trouble and expense to the members of keeping their books up to date.

The Administrative Committee has reached the definite conclusion that the loose-leaf and the loose-unit systems of publishing the Book of Standards are not practicable and that such advantages as they hold are greatly offset by many disadvantages, including increased expense over the present method. There is in the Executive Secretary's files a copy of the detailed analyses upon which this decision has been based.

The committee through its subcommittee also considered other proposals such as (1) staggering the publication of the Book of Standards so that one-third would appear each year; (2) using the compilations in place of the Book of Standards; and (3) publishing the Book of Standards—in five or more parts—as a binding together of individual compilations.

The committee's studies also indicated the possibility of publishing the standards in their present form but on an annual rather than triennial basis. More extensive study of this indicated that annual publication with receipts predicated on continuation of sales at present level would bring in nearly the same net revenue as the present scheme. The gross revenue would be greater with a new book on sale every year, but the outlay for printing would be $159,000 more over a six-year period. In the committee's opinion, it is more likely that sales would not exceed 75 per cent of their present rate, hence a net loss of revenue would accrue to the Society if annual publication were adopted. It accordingly did not pursue this proposal further. Neither did it consider the proposal to stagger the books to be desirable, since at no time would a complete book of reference then be available. Furthermore, the plan discussed below seemed to provide for spreading the load to almost an equal degree.

The committee is cognizant that in the present scheme of publishing the Book of Standards triennially there is some undesirable delay between adoption of new and revised standards (or tentatives) and their publication. In general, tentatives or standards adopted at the annual meeting seldom appear in the Book of Standards (or supplements thereto) earlier than the middle of December, and the publication has run as late as March. Hence, any scheme that might expedite publication would seem desirable.

At the present time, five volumes of A.S.T.M. standards are issued triennially in bound form and paper-bound supplements are issued during the second and third years. There are also now twenty-two compilations of selected standards which are issued annually, biennially, or triennially (although the present trend is towards annual publication). The portion of the present Books of Standards covered by these compilations is as follows:

Part IA, 1 compilation, approximately 25 per cent

Part IB, 4 compilations, approximately 75 per cent

Part II, 7 compilations, approximately 80 per cent

Part IIIA, 5 compilations, approximately 90 per cent

Part IIIB, 5 compilations, almost 100 per cent

The present method of publication necessitates repaging and double handling both at Headquarters and at the printer's on all standards which appear in both the compilations and the Book of Standards, which double handling involves more than 5000 pages. An estimate indicates that up to $5000 per issue could be saved in printing cost and three hundred man hours on the part of the staff if this double handling were eliminated—apart from expediting publication.

The fact that a considerable portion of the Book of Standards is now covered by compilations with an increasing trend on the part of the various committees to publish the standards under their jurisdiction in compilation form suggests that the compilations might be used as the basic form for the publication of standards and that the bound volumes could be then printed by overrunning the individual compilations. Such a scheme would involve binding together these overrun compilations of standards. Material that is duplicated in more than one compilation would be published in the back of the compilations so that the main portion of a compilation could be printed for Book of Standards and compilation use at one time and the remaining material run for only the compilation edition. Arrangements of standards within the compilations could either be on a classified basis or in numeric sequence. A general table of contents would appear in the front of the volume in which all standards and tentatives would be listed in the sequence in which they appear in the book as well as a table of contents arranged in numeric sequence although in either event the standards and tentatives would be merged, which is a departure from the present method of publishing the Book of Standards, in which they are segregated. A complete subject index would also be included at the back of the book to facilitate finding a given standard. On this basis the printing of the various volumes could be combined with existing compilations at less cost and considerable saving in time over the method now used.

Recommendations:

Specifically, the committee recommends first that the Book of Standards be issued in 1949 as six parts, as indicated in the accompanying Table I. This recommendation is premised on the fact that the present Part II is now approaching a size at which the amount of material contained therein is too bulky for economical binding or convenient handling. In order to indicate that each volume is a separate entity, it is suggested that parts be numbered from one to six rather than 1A, 1B, etc.

It is further recommended that each of the six parts be made up of overruns of individual compilations, much as indicated in Table I and as discussed above.

This latter proposal will save much repaging, as indicated, and will save considerable time at the printer's as well as staff time and should materially expedite publication of standards and compilations. At the present time with the duplicate paging required, compilations can only be issued early by delaying publ cation of the Book of Standards or *vice versa*, and to satisfy all members of the Society simultaneous publication

TABLE I.—SUGGESTED ARRANGEMENT OF 1949 BOOK OF STANDARDS.

PART 1.—FERROUS METALS

•Section A—Steel Pipe and Piping Materials (A-1)
Section B—Other Ferrous Standards
Section C—General Test Methods

PART 2.—NON-FERROUS METALS

•Section A—Electrical Resistance Materials (B-4)
•Section B—Copper and Copper Alloys (B-1 and B-5)
•Section C—Light Metals (B-7)
•Section D—Electrodeposited Coatings (B-8)
Section E—Other Non-Ferrous Standards
Section F—General Test Methods

PART 3.—CEMENTITIOUS, SOILS, ROADS AND WATERPROOFING MATERIALS

•Section A—Cement (C-1)
•Section B—Concrete and Non-Bituminous Road Materials (C-9 and D-4)
•Section C—Refractories (C-8)
•Section D—Glass (C-14)
Section E—Other cementitious, etc., Standards
•Section F—Bituminous Road and Waterproofing Materials (D-4 and D-8)
•Section G—Soils (D-18)
Section H—General Test Methods

PART 4.—PAINT, WOOD, ADHESIVES, SHIPPING CONTAINERS

•Section A—Paint and Naval Stores (D-1 and D-17)
Section B—Wood and Wood Preservatives (D-7)
•Section C—Adhesives
Section D—Shipping Containers
Section E—Fire Tests of Materials
Section F—General Test Methods

PART 5.—NON-METALLIC—FUELS, PETROLEUM, AROMATIC HYDROCARBONS, SOAP, WATER TEXTILES

•Section A—Petroleum Products and Aromatic Hydrocarbons (D-2 and D-16)
Section B—Gaseous Fuels (D-3)
•Section C—Coal and Coke (D-5)
•Section D—Soap (D-12)
•Section E—Textiles (D-13)
•Section F—Water (D-19)
Section G—General Test Methods

PART 6.—NON-METALLIC MATERIALS—ELECTRICAL INSULATION, PLASTICS, RUBBER, PAPER

•Section A—Electrical Insulating Materials (D-9)
•Section B—Plastics (D-20)
•Section C—Rubber Products (D-11)
•Section D—Paper and Paper Products (D-6)
Section E—General Test Methods

• Published also as compilations of Standards.

is desirable. This system also has the advantage that should annual publication be desirable, very little change will be necessary in publication practices and a minimum amount of staff work will be required because of the increasing trend for all standards to appear in compilation form.

Record of Publications Issued This Year

Apart from this very important subject of the scheme of publication, the activities of the Administrative Committee on Papers and Publications has been largely one of reviewing material that has been submitted for publication and in planning the technical program for the Annual Meeting. The remainder of this report is accordingly largely one of record of the various regular and special publications that have been issued during the past year. Mention might be made, however, of certain efforts that were made as an innovation to make the material available to the members more promptly. Most of the papers, for example, that had not been preprinted prior to the Annual Meeting were struck off as advance prints prior to the publication of the *Proceedings*. These were offered to the members on request at a nominal charge.

Regular Publications:

1947 Proceedings, 1207 pp, 7000 copies.
1947 Supplements to Book of Standards
  Part I-A, 421 pp, 10 000 copies
  Part I-B, 333 pp, 8 250 copies
  Part II, 483 pp, 8 000 copies
  Part III-A, 455 pp, 7 750 copies
  Part III-B, 323 pp, 7 250 copies
Index to 1947 Standards and Tentatives, 254 pp, 19 000 copies
Year Book, 542 pp, 5400 copies
ASTM BULLETIN, six issues, total number of pages 668, average number of copies 9375.

A statement of the volume of the principal publications for the past four years is given below:

Publications	1944, pages	1945, pages	1946, pages	1947, pages
Proceedings	1139	1060	1614	1206
Book of A.S.T.M. Standards	6042	....	6694	....
Supplements to Book of A.S.T.M. Standards	....	1189	....	2015
A.S.T.M. Methods of Chemical Analysis of Metals	32	....	412	....
Index to A.S.T.M. Standards and Tentatives	218	235	240	254
Year Book	408	474	512	542
Spring Meeting Symposiums	140	....	166	119
ASTM BULLETIN	464	492	600	668
	8443	3450	10238	4804

Special Compilations of A.S.T.M. Standards Published from April 15, 1947 to April 15, 1948:

Specifications for Steel Piping Materials (A-1), 316 pp, 2200 copies
Standards on Copper and Copper Alloys (B-5), 475 pp. 1200 copies
Standards on Cement (C-1), 200 pp, 2200 copies
Standards on Refractory Materials (C-8), 262 pp, 4400 copies
Standards on Paint, Varnish, Lacquer, and Related Products (D-1), 579 pp, 2000 copies
Standards on Petroleum Products and Lubricants (D-2), 706 pp, 7500 copies
Standards on Coal and Coke (D-5), 162 pp, 1600 copies
Standards on Electrical Insulating Materials (D-9), 588 pp, 1700 copies
Standards on Rubber Products (D-11), 571 pp, 1800 copies
Standards on Soaps and Other Detergents (D-12), 178 pp, 1500 copies
Standards on Textile Materials (D-13), 528 pp, 2600 copies
Standards on Adhesives (D-14), 48 pp, 1500 copies

Special Publications Also Issued:

Symposium on Testing of Parts and Assemblies, *STP 72*, 92 pp, 3000 copies
Symposium on pH Measurement, *STP 73*, 85 pp, 4000 copies
Symposium on Paint and Paint Materials, (1947) *STP 75*, 119 pp, 2000 copies
Symposium on Rubber Testing, *STP 74*, 115 pp, 2000 copies
Symposium on Synthetic Lubricants, *STP 77*, 36 pp, 3000 copies

- Petroleum Products as Related to Automotive Equipment, *STP 81*, 52 pp, 2000 copies
- 1947 Edgar Marburg Lecture—Engineering Laminates by W. C. Voss, 45 pp, 3000 copies
- 1946 Supplement to the Bibliography and Abstracts on Electrical Contacts (1946), *STP 56-C*, 27 pp, 1000 copies
- Report on Standard Samples for Spectrochemical Analysis, Compiled by E-2, Sub. IV (1947), *STP 58-A*, 29 pp, 2500 copies
- Radiographic Standards for Steel Castings, 50 sets of each
  - X-Ray Radiographic Standards—set of 31 plates
  - Gamma-Ray Radiographic Standards—set of 31 plates
  - 50 additional X-ray and 30 Gamma-Ray sets have been printed since the original printing in October.
- A.S.T.M. Manual of Engine Test Methods for Rating Fuels, 366 pp, 3000 copies (Several tables and certain charts referred to in the manual have been reprinted as separate copies or in the form of pads)

In addition to the above, the Symposium on Measurement of Entrained Air in Concrete has been reprinted from the 1947 *Proceedings*.

A partial Bibliography on Effect of Speed of Testing has been issued as a mimeographed document, 250 copies.

Reprints of the Publications Struck Off:

- Report of the Joint Committee on Standard Specification for Concrete and Reinforced Concrete, STP 5-A, 140 pp, 1500 copies
- Selected A.S.T.M. Standards for Students in Engineering, 294 pp, 11,917 copies—an additional 35,000 just printed and this contains some revisions. 292 pp.
- Procedures for Testing Soils, 200 pp, 1800 copies
- A.S.T.M. Manual on Presentation of Data, STP 15-B, 83 pp, 4000 copies
- Significance of Tests of Petroleum Products, STP 7-A, 40 pp, 1800 copies
- Evaluation of Petroleum Products, STP 45, 51 pp, 1000 copies
- Symposium on Lubricants, STP 29, 89 pp, 500 copies
- Viscosity Index Tables—STP 42, 35 pp, 2500 copies
- Viscosity Conversion Tables, STP 43, 12 pp, 2500 copies
- Viscosity Temperature Charts
  - Chart A: Saybolt Universal Viscosity, 20,000 copies
  - Chart B: Saybolt Universal Abridged, 16,000 copies
  - Chart C: Kinematic Viscosity, High Range, 10,000 copies
  - Chart D: Kinematic Viscosity, Low Range, 20,000 copies
  - Chart E: (new chart) Kinematic Viscosity, Low-Temperature Range, 10,000 copies

SPECIAL PUBLICATIONS IN PROSPECT

- Symposium on Effects of Low Temperatures on Materials—sponsored by the Philadelphia District at a meeting held in Philadelphia, March 1946. 100 pp, 4000 copies
- Symposium on Spectroscopic Light Sources—sponsored by Committee E-2 on Spectrographic Analysis and held at the annual meeting of the Society in June 1946. 120 pp, 2000 copies
- Symposium on Load Tests of Bearing Capacity of Soils—148 pp, 2500 copies
- 1947 Supplement to the Bibliography and Abstracts on Electrical Contacts, 32 pp, 1000 copies
- Manual on Fatigue Testing, 320 pp, (estimated), 3000 copies
- Tables of Data on Corrosion Resistance Steels (A-10), 75 pp, 4000 copies
- X-Ray Diffraction Data for Chemical Analysis, Second Supplement, 1500 cards, 750 sets

CURRENT ACTIVITIES

Program for the 1948 Annual Meeting:

A very full program is in prospect for the 1948 Annual Meeting with a number of symposiums and sessions scheduled for presentation as follows:

- Symposium on Magnetic Testing—under the auspices of Committee A-6 on Magnetic Properties
- Symposium on Usefulness and Limitations of Samples—under the auspices of Committee E-11 on Quality Control of Materials
- Symposium on Metallography in Color—under the auspices of Committee E-4 on Metallography
- Panel Discussion on The Influence of Non-Ferrous Metals and Their Compounds on the Corrosion of Pressure Vessels—under the joint auspices of Committee D-19 on Water for Industrial Uses and the Joint Committee on Boiler Feed-water Studies
- Symposium on Speed of Testing—under the auspices of Committee E-1 on Methods of Testing

Symposium on Functional Tests for Ball-Bearing Greases—under the auspices of Committee D-2 on Petroleum Products

Symposium on Mineral Aggregates—under the joint auspices of Committees C-9 on Concrete and D-4 on Road and Paving Materials

Session sponsored by Jt. Committee on Effect of Temperature

Symposium on Deformation of Metals as Related to Forming and Service—under the auspices of the Administrative Committee on Simulated Service Testing

Symposium on Test Methods and Procedures Used in Identifying Reactive Materials—under the auspices of Committee C-9 on Concrete

In addition to the above there are a number of other technical papers not a part of any formal symposium.

A number of papers are also being offered to the Society for publication but not presentation at a meeting. These for the most part are published in the ASTM BULLETIN although in some cases such papers are preprinted separately and subsequently published in the *Proceedings*.

Respectfully submitted on behalf of the committee,

C. L. Warwick,
Chairman

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON ULTIMATE CONSUMER GOODS

During the past year the Administrative Committee on Ultimate Consumer Goods has held one meeting which took place on March 30, 1948, at Society Headquarters, Philadelphia, Pa. All members were present.

In the report of the committee a year ago, mention was made that in taking cognizance of the inter-action of the physical and social sciences in the development of test methods and specifications for ultimate consumer goods, there had been organized two task groups to assist the Administrative Committee in formulating a suitable program. The one task group, under the chairmanship of Prof. C. West Churchman, formerly located at the University of Pennsylvania, and now at Wayne University, Detroit, Mich., was to work in the area of that which makes a thing wantable in terms of characteristics that make it satisfactory, adequate, dependable, or economic to some one or more persons. The other task group, under the Chairmanship of Prof. J. C. Whitwell of Princeton University, was to cover the area of that which characterizes a thing itself independent of all other things and of human volition and interest. It was planned to present research programs emanating from these two task groups to the Committee on Measurement of Opinion, Attitudes and Consumer Wants, which was organized jointly by the National Research Council and the Social Science Research Council, through C. L. Warwick, the A.S.T.M. representative. Reports from these two groups were received during the year, Dr. Paul S. Olmstead, Bell Telephone Labs., serving as acting Chairman of Professor Whitwell's group. These reports discussed the programs and problems before these two groups and it is proposed to have articles prepared based on these reports for publication in the A.S.T.M. BULLETIN.

While the two task groups have been discharged upon the completion of their assignments, the Administrative Committee plans to form an advisory group of social scientists, under the chairmanship of Dr. Olmstead, to keep the Administrative Committee advised and oriented on the progress which is made in the attack upon the problems related to consumer want determination.

Mention was also made in the report a year ago that consideration was being given to the procedure that should be recommended to technical committees for use in developing standards for consumer goods. Such standards will not carry proper weight if in their establishment there has not been adequate representation from those groups which are competent to express the viewpoint of the ultimate consumer. A revision of the Regulations Governing Technical Committees to take care of this matter has been recommended to the Board of Directors.

It is recommended that Messrs. A. G. Ashcroft, H. J. Ball, and G. C. MacDonald, whose terms of membership on the Administrative Committee expire in June, 1948, be reappointed for an additional term of three years. The Administrative Committee also recommends that one or two additions be made to the committee.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

Although the committee has held but one formal meeting—and one informal session—during the year, a number of the previously initiated actions have been successfully concluded, and others inaugurated at the January 21, 1948, meeting.

Of the older items, the present situation in the annular bearing field was considered to be in sufficiently good status as not to warrant the formation of the proposed joint committee with the Annular Bearing Engineers' Committee. On the other hand, the consideration of sandwich type materials has resulted in the formation of Committee C-19 on Structural Sandwich Constructions which held its organization meeting on January 30, 1948.

While the situation in the annular bearing field is quite good, the sleeve bearing field is in need of a considerable amount of assistance along lines which involve several A.S.T.M. Committees concerned with the bearing materials, lubricants, oxidation, corrosion, etc. In addition, a general classification of sleeve type bearing materials on the basis of "intended" uses is highly desirable.

As the result of A.C.S.S.T. consideration of this subject, it was decided to sponsor—through the cooperation of the several committees concerned—a symposium on "Bearing Metals and Their Lubricants," tentatively scheduled for the June, 1949, annual meeting.

The subject of humidity testing has been under consideration by this committee and activity is now in progress to correlate the efforts in the several committees concerned in an attempt to arrive at a set of test conditions which will permit the establishment of one or more standard humidity tests. Some work in this direction seems to be progressing satisfactorily at Wright Field, and it is possible that the methods and equipment used on this project may form the basis of the proposed standard tests.

It is the consensus of the Administrative Committee that the present status of the methods used for studying metal deformation, flow, etc., and the interpretations of results have now reached the stage where much wider attention should be given to these procedures as well as to new potential methods of test which will go much further than many of our present methods in giving a basic evaluation of properties. To this end, the committee is sponsoring a symposium at the June, 1948, meeting which covers the testing aspects of these new developments in fields ranging from ships to aircraft, and involving a number of different materials. Six papers are scheduled for this presentation, all of which will endeavor to emphasize the superiority of the newer test procedures and their interpretations.

A number of items, including such applications as gas turbine materials, soldered joints, decarburization, vibration, abrasion, etc., have been considered and referred to other A.S.T.M. Committees who are, or might be, concerned with such activities.

There are no specific recommendations to the Board of Directors at this time.

Inasmuch as the Board of Directors will act this spring on appointments to the committee, it was considered advisable to postpone the election of officers until after the Board action.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

C. S. COLE,
Secretary.

APPENDIX VII

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

Including the Lower Ohio Valley District now in course of organization, there are at present 13 formally organized A.S.T.M. districts. Functioning under district councils, the personnel of which are nominated and elected by the various districts, these groups have completed their first full year of operation under the revised district Charter and Manual. The Administrative Committee is gratified with the manner in which the councils have administered their respective district affairs and wishes to compliment the district officers on their initiative and close cooperation.

The new district centering in Washington, D. C., was formally organized and is now planning a 1948–1949 program. The Lower Ohio Valley District is in process of nominating officers. The group of councilors *pro tem* appointed by the Board of Directors took advantage of meetings of technical committees in February and held the first meeting in Dayton, a very successful one. President Boyd and Administrative Committee Chairman Fellows were present.

In the May, 1948, A.S.T.M. BULLETIN (page 45) there is shown a map of the United States with the new district areas designated. Practically all districts, except the two in California, were considerably expanded, and all of the north eastern and north central portions of the United States have been covered, including the heaviest concentration in Ontario, Can. About 85 per cent of the entire membership in the United States is now encompassed by some district.

The 1947 Year Book anticipated the expanded districts, and in the section on geographical distribution of members, states and localities are keyed with the respective district numbers.

Several of the districts have cooperated in membership promotion, particularly along so-called follow-up lines, and committees are being appointed in the districts to work with the Society's main Membership Committee. The Southern and Northern California districts plan intensive work on the west coast. Membership promotion is one activity where district councils and, in fact, all district members can help greatly.

The Administrative Committee held one meeting in June, 1947, and will meet in Detroit in June, 1948. The committee will again sponsor a breakfast meeting of district officers at which those responsible for guiding district affairs and the officers of the Society can exchange a few ideas.

The Administrative Committee plans to consider several matters affecting the welfare of districts and the Society. One of these is the question of topics to be covered at various district meetings. Some integrated program may be desirable and helpful. Another subject is the desirability of organizing districts in areas where there is considerably less concentration of members than is considered sufficient for normal district operations. The Administrative Committee is cognizant of the fact that some local activities are desirable in knitting our members more closely together.

Through its deliberations and suggestions, the Administrative Committee wants sincerely to lend its fullest support to the respective district activities and to stimulate those which are in the best interests of the Society and its membership and of the groups which A.S.T.M. serves.

Respectfully submitted on behalf of the committee,

C. H. FELLOWS,
Chairman.

R. J. PAINTER,
Secretary.

REPORT OF COMMITTEE A-1*
ON
STEEL

While Committee A-1 has been active in the preparation of specifications for low-temperature applications, there has been quite a bit of other activity among the subcommittees in formulating new specifications and methods and in revising existing documents to keep abreast of changing industrial practice.

It is of interest to note that Subcommittee XXII has discharged its Section on the Properties of Metals at Subatmospheric Temperatures and appointed a Low-Temperature Advisory Committee with E. O. Dixon as chairman. This group will work in cooperation with the other sections of Subcommittee XXII, giving advice on the development of specifications and methods of test for low-temperature service materials.

Personnel.—The committee membership still continues to grow with, at present, 273 voting members, consisting of 133 producers, 106 consumers, and 34 general interest members. C. E. Loos of the Carnegie-Illinois Steel Corp. has been appointed vice-chairman of Subcommittee II on Structural Steel.

Subsequent to the resignation in 1947 of E. J. Edwards, American Locomotive Co., from the chairmanship of Subcommittee XI on Steel for Boilers and Pressure Vessels, H. G. Miller, Chicago, Milwaukee, and St. Paul Railroad has accepted an appointment to this position and W. Warner, Inland Steel Co., will act as secretary of Subcommittee XI. In recognition of his long-time service to Subcommittee XI, Committee A-1 at its June, 1947, meeting tendered E. J. Edwards a formal vote of thanks.

Losses by Death.—The Steel Committee has suffered the loss through death of several members who took a most active interest in its work. Two of these men, C. F. W. Rys, long-time Metallurgical Engineer of the Carnegie-Illinois Steel Corporation and its predecessor, and Allan W. Carpenter, Engineer of Bridges, New York Central Railroad, were active in many phases of the work over long periods of time. The work of these men in behalf of Committee A-1 had been recognized by their election as honorary A-1 members. Mr. B. L. McCarthy, Wickwire Spencer Steel Co., who also died, was particularly active in the work on spring wire.

The Committee records its appreciation of the services and constructive interest of these men.

TENTATIVES AND REVISIONS OF TENTATIVES APPROVED DURING THE PAST YEAR

Subsequent to the 1947 Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Seamless 1 Per Cent Chromium, 0.5 Per Cent Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315 - 47 T).

Revision of Tentative Specifications for:

Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 - 44 T),

Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 - 44 T),

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

Alloy-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217 – 46 T),

Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 46 T),

Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 46 T) and

Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperature (A 280 – 46 T).

Tentative Revision of Standard Specifications for:

Steel for Bridges and Buildings (A 7 – 46), and

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 46).

Revision and Reversion to Tentative of Standard Specifications for:

Carbon-Steel Axles for Cars and Tenders (A 21 – 47).

These recommendations were accepted by the Standards Committee on February 6, 1948, and the new and revised tentatives together with the tentative revisions appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

On February 16, 1948, the Standards Committee accepted the Tentative Specifications for Stress-Relief-Annealed Cold-Drawn Carbon-Steel Bars (A 311 – 47 T). The new tentative appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

The proposed Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service were accepted as tentative by the Standards Committee on April 23, 1948. The new tentative specifications have been assigned the designation A 320 – 48 T.

The committee has also approved the Proposed Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe[1] prepared by a joint Committee A-1 and Committee A-10 task group.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending for publication as tentative two new methods of testing as appended to this report. Revisions in eight tentatives and seven standards are recommended as itemized in the Appendix to this report. A tentative revision is recommended for one standard and one standard is being reverted to tentative with revision. Editorial corrections in four specifications are recommended.

These actions have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Steel Rails and Accessories:

Two sections of this subcommittee met in Philadelphia during the week of February 2. The Section on Rail Joint Bars and Track Bolts proposed revisions in Standard Specifications A 5 and A 49 as outlined in the Appendix to this report. The Section on Tie Plates recommended certain desirable changes in Specifications A 67 and A 241 to parallel the existing A.R.E.A. documents and to incorporate editorial changes, as outlined in the Appendix.

Subcommittee II on Structural Steel for Bridges, Buildings and Rolling Stock:

Significant work is being carried out by this subcommittee involving the development of a new specification containing all appropriate tables and tolerances of material common to the various structural specifications, which would then be deleted from the present existing documents and covered by reference only. Subcommittee XI is interested in this development since a similar one could be worked out for the boiler steel specifications.

Proposed revisions in Specifications A 284 involving, among other considerations, the adoption of a low strength grade, are under study.

---

[1] See Editorial Note, p. 94.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee IV on Spring Steel and Steel Springs:

This subcommittee has developed and submitted to Subcommittee XIII a proposed Tentative Method of Tension Testing of Steel Spring Wire.

It has also developed a Proposed Method of Twist Testing of Steel Spring Wire which is being submitted to Subcommittee XIII for its approval. In addition, various editorial changes in Specifications A 125, A 147, A 61 and A 62 have been approved by Committee A-1 as outlined in the Appendix to this report.

Subcommittee VI on Steel Forgings and Billets:

Subcommittee VI held a meeting in Philadelphia during the week of February 2 and also a special meeting in Philadelphia on April 20. Intensive work is being done on revisions in the various specifications under the jurisdiction of this subcommittee to bring them up to date with manufacturing practices and in line with one another. Specifications A 235, A 236, A 237, A 238, A 243 and A 266 will be reverted to a tentative status, so the revision work may be more easily handled. Tentative Specifications A 288, A 289, A 290, A291, A 292, A 293, and A 294 will also be revised.

Subcommittee VII on Rolled Steel Wheels and Steel Tires:

This subcommittee recommends a change in the chemical requirements of Specifications A 25 as given in the Appendix to this report. It also recommends various editorial changes in Specifications A 57 as outlined.

Subcommittee VIII on Steel Castings:

Tentative Methods A 272 have been reviewed by the subcommittee, and it has been decided to continue the methods as tentative. Certain desirable editorial changes are under consideration and also higher silicon limits in Tentative Specifications A 27.

Subcommittee IX on Steel Tubing and Pipe:

A joint group of Subcommittee IX and Subcommittee XXII personnel has drafted two specifications for low-temperature application, one for pipe and one for tubing. Both specifications have been referred to the Low-Temperature Advisory Section of Subcommittee XXII for study and recommendations. Tentatively the tubing specification will be under the jurisdiction of Subcommittee IX and the pipe specification under Subcommittee XXII.

The subcommittee is recommending a tentative revision to Standard Specifications A 139 as covered in the Appendix.

Specifications A 181 on Forged or Rolled Steel Pipe Flanges for General Service are now under the jurisdiction of Subcommittee XXII, following recommendation by Subcommittee IX which has been approved by the A-1 Advisory Committee.

Subcommittee XI on Steel for Boilers and Pressure Vessels:

While this subcommittee has no immediate recommendations for revisions there is a large volume of work under way covering revisions in the existing specifications. Also, two new documents are under consideration including a specification for $8\frac{1}{2}$ per cent nickel steel plate and a master specification similar to the one being developed by Subcommittee II.

A new chairman and a secretary have been appointed for Subcommittee XI as covered previously in the report.

Subcommittee XIII on Methods of Physical Tests:

Subcommittee XIII has approved the

Proposed Tentative Method of Macroetch Testing of Forgings, and Blooms and Billets Intended for Forgings,[3] submitted by Subcommittee VI, with the understanding that this method is intended only for forgings, and blooms and billets intended for forgings, and is not intended as being necessarily applicable to other products without review by the specific product subcommittee. To clarify the intent of Subcommittee VI that the individual specifications for which the method is intended should carry a reference to the method, Subcommittee XIII revised the scope clause to read as follows by the addition of the italicized words:

> This method of macroetch testing and inspection is intended to apply to steel forgings and also to the billets and blooms intended to be used as stock for the manufacture of forgings *when this method of testing is referred to by a given specification.*

A Proposed Tentative Method of Tension Testing of Steel Spring Wire,[3] submitted by Subcommittee IV has also been approved. Subcommittee XIII is cognizant of the fact that valuable procedures for tension testing appear in Methods E 8, but feels that the methods proposed by product subcommittees of Committee A-1 are intended to apply definitely to individual cases for which they are designed in more specific detail than would appear practical in Method E 8.

Subcommittee XV on Bar Steels:

A new Proposed Specifications for Heat-Treated Carbon-Steel Bars written by this subcommittee has been approved by Committee A-1. Subsequent to the incorporation in these specifications of certain editorial corrections, it will be submitted to the Administrative Committee on Standards for approval.

Also under consideration by this subcommittee is a new Proposed Specifications for Alloy-Steel Bars to Chemical Requirements.

Subcommittee XIX on Sheet Steel and Steel Sheets:

A Proposed Specification for Sheets of Flange and Firebox Quality is under consideration by Subcommittee XIX. After certain changes have been effected the document will be submitted to Subcommittee XI for criticism and suggestions.

Specifications A 245, A 246, and A 303 are being revised as described in the Appendix.

Subcommittee XXII on Valves, Fittings, Piping and Flanges for High Temperature and Subatmospheric Temperatures:

The two new Specifications A 315 and A 320, referred to earlier in this report, originated in this subcommittee.

A group has been formed to prepare a specification for forgings for low-temperature service. The jurisdiction of Specifications A 181 has been transferred from Subcommittee IX to this subcommittee as described previously in this report.

Specifications A 182 is being heavily revised and reverted to a tentative status as outlined in the Appendix.

A Proposed Specification for Pipe for Low-Temperature Service is under consideration by the subcommittee, with the Section on Pipe and the Low-Temperature Advisory Section participating. Specifications A 106, A 158, A 194, A 206, A 280 and A 315 are being revised as outlined in the Appendix.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, N. L. Mochel.
Vice-Chairman, H. B. Oatley.
Vice-Chairman, T. G. Stitt.
Secretary, H. C. Larson.

---

[3] These methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

This report has been submitted to letter ballot of the committee, which consists of 273 members; 176 members returned their ballots, of whom 164 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

HENRY WYSOR,
Secretary.

EDITORIAL NOTE

On November 15, 1948, the Administrative Committee on Standards accepted the joint recommendation of Committees A-1 and A-10 that the Specifications for Seamless and Welded Austenitic Stainless Steel Pipe be approved for publication as tentative. The new tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A, bearing the designation A 312 – 48 T.

The following recommendations, submitted to the Administrative Committee on Standards by Committee A-1, were approved on December 22, 1948:

Revision of Tentative Specifications for:

Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 48 T).

Tentative Revision of Standard Specifications for:

Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 48).

The revised tentative specifications as well as the tentative revision appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

On December 23, 1948, the Standards Committee accepted revisions of the Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 47 T). The revised tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

The proposed revisions of the Tentative Method of End-Quench Test for Hardenability of Steel (A 255 – 46 T) were accepted by the Standards Committee on January 3, 1949. The revised tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in either the 1946 Book of A.S.T.M. Standards, Part I-A, or the 1947 Supplement, Part I-A, as indicated by the final number in the A.S.T.M. Designation.

Revisions in High-Temperature Pipe Specifications—Weight Requirements—Finish Clauses.—In the following high-temperature pipe specifications: namely,

A 106 - 47 T
A 158 - 47 T
A 206 - 47 T
A 280 - 47 T
A 315 - 47 T

revise the requirements for weighing the pipe to read as follows:

> Unless otherwise agreed upon between the manufacturer and the purchaser, pipe in sizes 4 in. and smaller may be weighed in convenient lots; pipe in sizes larger than 4 in. shall be weighed separately.

The previous wording permitted pipe in sizes smaller than 4 in. to be weighed in convenient lots.

Also in Specifications A 158, A 206, A 280, and A 315 revise the section on finish to read as follows:

> (*a*) The finished pipe shall be reasonably straight and free from injurious defects. At the discretion of the inspector representing the purchaser, finished pipe shall be subject to rejection if surface defects acceptable under Paragraph (*c*) are not scattered, but appear over a large area in excess of what is considered a workmanlike finish.
>
> (*b*) *Depth of Injurious Defects.*—All defects shall be explored for depth. When the depth is in excess of $12\frac{1}{2}$ per cent of the nominal wall thickness or encroaches on the minimum wall thickness, such defects shall be considered injurious.
>
> (*c*) *Machining or Grinding Defects not Classified as Injurious.*—Surface defects not classified as injurious shall be treated as follows:
>
> (*1*) Pipe showing scabs, seams, laps, tears, or slivers not deeper than 5 per cent of the nominal wall thickness need not have these defects removed. If deeper than 5 per cent, such defects shall be removed by machining or grinding.
>
> (*2*) Pipe showing inside or outside surface checks (fish scale) 1/64 in. or less in depth need not have these defects removed. Such defects over 1/64 in. but not more than 1/32 in. in depth shall be removed by machining or grinding. Pipe on which these defects are more than 1/32 in. in depth shall be rejected, unless the manufacturer can demonstrate to the purchaser that the defects are not injurious as defined in Paragraph (*b*).
>
> (*3*) Mechanical marks or abrasions[a] and pits shall be acceptable without grinding or machining provided the depth does not exceed the limitations set forth in Paragraph (*b*) and if not deeper than $\frac{1}{16}$ in. If such defects are deeper than $\frac{1}{16}$ in. but not deeper than $12\frac{1}{2}$ per cent of the nominal wall thickness, they shall be removed by grinding or machining to sound metal.
>
> (*4*) When defects have been removed by grinding or machining, the outside diameter at the point of grinding or machining may be reduced by the amount so removed. Should it be impracticable to secure a direct measurement, the wall thickness at the point of grinding, or at a defect not required to be removed, shall be determined by deducting the amount removed in grinding or the depth of the defect, from the minimum measured wall thickness at the ends of the pipe, and the remainder shall not be less than $87\frac{1}{2}$ per cent of the nominal wall thickness.
>
> (*5*) Machining or grinding shall follow the inspection at the mill of the pipe as rolled and shall be followed by supplementary visual inspection.

---

[a] **Marks and abrasions arede fined as cable makrs, dinges, guide marks, roll marks, ball scratches, scores, etc.**

(*d*) *Repair by Welding.*—Repair of injurious defects shall be permitted only subject to the approval of the purchaser and with the further understanding that the composition of the welding rod shall be suitable for the composition of the metal being welded. Welding of injurious defects in no case shall be permitted when the depth of defect exceeds $33\frac{1}{3}$ per cent of the nominal pipe wall thickness or the length of repair exceeds 25 per cent of the nominal diameter of the pipe. Defects shall be thoroughly chipped out before welding, and then heat treated in accordance with Section 4. Each length of repaired pipe shall be retested hydrostatically in accordance with Section 11.

REVISIONS OF TENTATIVES

Tentative Specifications for Heavy Gage Structural Quality Flat Hot-Rolled Carbon-Steel Sheets (A 245 – 47 T):

Section 1.—Add the following to this section: "Flat hot-rolled carbon-steel may be spot welded in the "hot-rolled" condition, but for best results the oxide or scale should be removed before welding."

Section 4.—Add the following as a footnote to the table in this section—reference after the maximum carbon content of 0.25 per cent for open-hearth steel: "As carbon increases above 0.15 per cent spot welding becomes increasingly difficult."

Section 7 (*b*).—Delete the words "use of the."

Section 8.—For grade C material change the bend test requirements to read as follows:

> The bend test specimen shall stand being bent at room temperature in any direction through 180 deg. around one and one-half thicknesses of the material, without cracking on the outside of the bent portion.

Table II.—Revise footnote *b* to read as follows:

> The tonnage ordered for shipment to one place at one time determines the producer's responsibility for weight variation for that tonnage. Any partial shipment against that tonnage should not vary more than the percentage given in the table for the lesser amount.

Table III.—The column covering permissible variations from thickness for specified widths and thicknesses of 0.1874 to 0.1420 in., incl., shall be revised to read as follows:

Specified Width, in.	0.1874 to 0.1800 in., incl.	0.1799 to 0.1420 in., incl.
Over 12 to 15, incl.	0.007	0.007
Over 15 to 20, incl.	0.008	0.008
Over 20 to 32, incl.	0.009	0.009
Over 32 to 40, incl.	0.009	0.009
Over 40 to 48, incl.	0.010	0.010
Over 48 to 60, incl.	.....	0.010
Over 60 to 70, incl.	.....	0.011
Over 70 to 80, incl.	.....	0.012
Over 80 to 90, incl.	.....	0.012
Over 90	.....	....

Section 11.—Revise Paragraph (*a*) to read as follows: "When sheets are ordered to weight (gage numbers), the weight shall not vary from those specified more than the amounts shown in Table II."

TABLE I.—PERMISSIBLE VARIATIONS IN LENGTH.

(Sheets Not Resquared)

Specified Lengths, in.	Variation Over Specified Lengths, in.[a]
To 15, incl.	1/8
Over 15 to 30, incl.	1/4
Over 30 to 60, incl.	1/2
Over 60 to 96, incl.	3/4
Over 96 to 120, incl.	1
Over 120 to 156, incl.	1 1/4
Over 156 to 192, incl.	1 1/2
Over 192 to 240, incl.	1 3/4
Over 240	2

[a] No permissible variation under.

Revise Paragraph (*e*) and the note to read as follows:

> (*e*) The permissible camber on hot-rolled sheets shall not exceed the amounts shown in Table VI.
>
> NOTE.—Camber is the greatest deviation of a side edge from a straight line; and measurement is taken on the concave side with a straight edge.

Table IV.—Delete the tolerances for specified widths "2 and under" and "Over 2 to 6, incl." The present tolerances for specified widths "Over 6 to 15, incl." shall be applied for widths "Up to 15, incl."

Table V.—Revise Table V to read as

shown in the accompanying Table I.

New Table.—Insert a new Table VI to read as shown in the accompanying Table II, renumbering the present Table VI as Table VII.

New Appendix II.—Add a new Appendix II on "Recommended Tentative Standard Thicknesses for Hot-Rolled Carbon-Steel Sheets" to read as follows:

> General: The thicknesses proposed are for all uncoated metals and alloys, and are based on the 20-series of preferred numbers rounded off to the third decimal place.

A table of preferred thicknesses as shown in the accompanying Table III will then follow the text material.

TABLE II.—PERMISSIBLE CAMBER ON HOT-ROLLED SHEETS.

Sheet Length, ft.[a]	Permissible Camber, in.
To 4, incl.	⅛
Over 4 to 6, incl.	³⁄₁₆
Over 6 to 8, incl.	¼
Over 8 to 10, incl.	⁵⁄₁₆
Over 10 to 12, incl.	⅜
Over 12 to 14, incl.	½
Over 14 to 16, incl.	⅝
Over 16 to 18, incl.	¾
Over 18 to 20, incl.	⅞
Over 20 to 30, incl.	1¼
Over 30 to 40, incl.	1½

[a] For sheets in coils, permissible camber shall not exceed 1 in. in any 20 ft. of length.

TABLE III.—PREFERRED THICKNESSES FOR UNCOATED METALS AND ALLOYS.

	0.1800	0.0900	0.0450	0.0220	0.0110	
	0.1600	0.0800	0.0400	0.0200	0.0100	0.0050
	0.1400	0.0710	0.0360	0.0180	0.0090	
	0.1250	0.0630	0.0320	0.0160	0.0080	0.0040
0.2240	0.1120	0.0560	0.0280	0.0140	0.0070	
0.2000	0.1000	0.0500	0.0250	0.0120	0.0060	

Tentative Specifications for Light Gage Structural Quality Flat Hot-Rolled Carbon-Steel Sheets (A 246 – 47 T):

Section 1.—Revise as indicated above for Section 1 in Specifications A 245.

Section 4.—Revise as indicated above for Section 4 in Specifications A 245.

Section 7 (b).—Revise as indicated above for Section 7(*b*) in Specifications A 245.

Section 8.—Revise as indicated above for Section 8 in Specifications A 245.

Table II.—Revise as indicated above for Table II in Specifications A 245.

Section 11.—Revise Paragraph (*a*) as indicated above for Section 11(*a*) in Specifications A 245.

Revise Paragraph (*e*) and the note to read as follows:

> (*e*) The permissible camber shall not exceed the amounts given in Table VI.
>
> NOTE.—Camber is the greatest deviation of a side edge from a straight line; and measurement is taken on the concave side with a straight edge.

Table IV.—Revise as indicated above for Table IV in Specifications A 245.

New Table.—Add a new Table VI to read as shown in the accompanying Table II.

New Appendix.—Add a new Appendix II to read as shown for the new Appendix II in Specifications A 245.

Tentative Specifications for Hot-Rolled Strip of Structural Quality (A 303 – 47 T):

Section 1.—Revise as indicated above for Section 1 in Specifications A 245.

Section 3.—Revise as indicated above for Section 4 in Specifications A 245.

Section 6.—Revise as indicated above for Section 7 in Specifications A 245.

Section 7.—Revise as indicated above for Section 8 in Specifications A 245.

Section 10.—Revise Paragraph (*a*) by adding the following sentence: "Strip shall not vary from the specified thickness at the center (crown) more than the edge tolerance in Table II plus the amount shown in Table III."

Revise the table in Paragraph (*d*) to read as follows:

For strip over 1½ in. in width	¼ in. in any 8 ft.
For strip 1½ in. and under in width	½ in. in any 8 ft.

> NOTE.—Camber is the greatest deviation of a side edge from a straight line; and measurement is taken by placing an 8-ft. straight edge on the concave side and measuring the distance between the strip edge and the straight edge in the center of the arc.

Table II.—Revise footnote *a* to read

as follows: "Thickness measurements for Table II are taken $\frac{3}{8}$ in. from edge on strip of 1 in. or wider; and at any place on the strip when narrower than 1 in. The given variations do not include crown."

Table III.—Delete the present crown tolerances for a thickness of 0.2299 to 0.2031 in., incl., in widths over 1 to 6 in., incl. For thicknesses 0.1874 to 0.1180 in., incl., in widths of over 1 to $3\frac{1}{2}$ in., incl., change the present crown tolerance of "0.001" to "0.002" in.

New Table.—Insert a new Table IV to read as shown in the accompanying Table IV.

TABLE IV.—PERMISSIBLE VARIATIONS IN WIDTH.

Ordered Width, in.	Permissible Variations from Ordered Width for Thicknesses Given, Over or Under, in.		
	Mill Edge and Square Edge, All Thicknesses	Slit Edge	
		To 0.109, incl.	Over 0.109
To 2, incl.	$\frac{1}{32}$	0.008	0.016
Over 2 to 5, incl.	$\frac{3}{64}$	0.008	0.016
Over 5 to 10, incl.	$\frac{1}{16}$	0.010	0.016
Over 10 to 12, incl.	$\frac{3}{32}$	0.016	0.016

Table V.—Renumber as Table VI and change the last line of this table from the present "Over 6" to read "Over 6 to 12, incl."

New Appendix.—Add a new Appendix to these specifications entitled "Recommended Tentative Standard Thicknesses for Hot-Rolled Carbon-Steel Strip," the Appendix to read as indicated above for the new Appendix II in Specifications A 245.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 46):

Tables I and II.—Delete grades F 3, F 4, F 7, F 11, and F 12.

Revise the chemical requirements of the remaining grades and add new grades so that the tables read as shown in the accompanying Tables V and VI.

TENTATIVE REVISION OF STANDARD

Standard Specifications for Electric-Fusion Welded Steel Pipe (Sizes 4 in. to but not Including 30 in.) (A 139 – 46):

Section 14 (a).—Revise the requirement for a bevel of "37.5 deg." to "35 ± 2.5 deg."

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for High Carbon Steel Joint Bars (A 5 – 39):

Section 14.—Revise to read as follows:

14. *Marking and Stamping.*—The name or brand of the manufacturer, the section designation, and the year of manufacture shall be rolled in raised letters and figures on the side of the rolled bars and a portion of this marking shall appear on each finished joint bar. A serial number representing the heat or melt shall be hot stamped on the outside of the web of each bar, near one end.

Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 – 46):

Section 4.—Revise the requirement for carbon content from the present "0.65 to 0.85" to read "0.65 to 0.80" per cent.

Standard Specifications for Quenched Carbon-Steel Joint Bars (A 49 – 39):

Section 15.—Revise to read as indicated above for Section 14 in Specifications A 5.

Standard Specifications for Multiple-Wear Wrought Steel Wheels (A 57 – 46):

Section 4 (b).—Revise the second sentence to read as follows by the deletion of the word in brackets: "The cooling shall be controlled to prevent injury by too rapid cooling [to] below the critical range."

TABLE V.—CHEMICAL REQUIREMENTS.

Type	Ferritic Steels					
Identification Symbol	F 1	F 5	F 17	F 6	F 22	F 2
Grade	Carbon-Molybdenum	4 to 6 per cent Chromium	9 per cent Chromium	13 per cent Chromium	Chromium-Molybdenum	1 per cent Chromium, 0.5 per cent Molybdenum
Carbon, per cent	0.20 to 0.30	0.25 max.	0.15 max.	0.12 max.	0.15 max.	0.10 to 0.20
Manganese, per cent	0.60 to 0.80	0.60 max.	0.30 to 0.60	1.00 max.	0.30 to 0.60	0.30 to 0.80
Phosphorus, max., per cent	0.040	0.040	0.030	0.040	0.030	0.040
Sulfur, max., per cent	0.040	0.030	0.030	0.030	0.030	0.040
Silicon, per cent	0.20 to 0.35	0.50 max.	0.50 to 1.00	1.00 max.	0.50 max.	0.10 to 0.60
Nickel, per cent	....	0.50 max.	....	0.50 max.	....	
Chromium, per cent	....	4.0 to 6.0	8.0 to 10.0	11.5 to 13.5	2.0 to 2.50	0.80 to 1.10
Molybdenum, per cent	0.40 to 0.60	0.45 to 0.65	0.90 to 1.10	....	0.90 to 1.10	0.45 to 0.65

Type	Austenitic Steels				
Identification Symbol	F 8	F 8m	F 8c	F 10	F 25
Grade	18 Chromium, 8 Nickel	18 Chromium, 8 Nickel Modified with Molybdenum	18 Chromium, 8 Nickel Modified with Columbium	20 Nickel, 8 Chromium	A.I.S.I. 310
Carbon, per cent	0.08 max.	0.08 max.	0.08 max.	0.10 to 0.20	0.15 max.
Manganese, per cent	2.00 max.	2.00 max.	2.00 max.	0.50 to 0.80	2.00 max.
Phosphorus, max., per cent	0.040	0.040	0.040	0.030	0.040
Sulfur, max., per cent	0.030	0.030	0.030	0.030	0.030
Silicon, per cent	1.00 max.	1.00 max.	1.00 max.	1.00 to 1.40	1.00 max.
Nickel, per cent	8.00 to 11.00	10.00 to 14.00	9.00 to 12.00	19.00 to 22.00	19.00 to 22.00
Chromium, per cent	18.00 to 20.00	16.00 to 18.00	17.00 to 19.00	7.00 to 9.00	24.00 to 26.00
Molybdenum, per cent	....	2.00 to 3.00	....	....	....
Columbium, per cent	....	....	[a]	....	....

[a] Grade F 8c shall have a columbium content of not less than ten times the carbon content and not more than 1.00 per cent.

Section 13 (r).—Revise to read as follows:

> Where dimension R_1 or R_2 is marked "f" extra stock of $\frac{1}{16}$ to $\frac{3}{16}$ in. may be left for finish or wheel may be furnished finished to exact dimension shown. Hub length tolerance is based on finish dimension.

Standard Specifications for Steel Tie Plates (A 67 – 46):

Title.—Change to read as follows "Standard Specifications for Low Carbon Steel Tie Plates."

Section 1.—Revise to read "These specifications cover low-carbon steel tie plates for use in railroad rails."

Section 3.—Revise the table of chemical requirements to read as follows:

	Acid-Bessemer	Open-Hearth or Electric-Furnace
Carbon, min., per cent	0.08	0.15
Phosphorus, max., per cent	0.11	0.05
Copper, when copper steel is specified, min., per cent	0.20	0.20

TABLE VI.—TENSILE REQUIREMENTS.

Grade	Tensile Strength, min., psi.	Yield Point, min., psi.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
F 1	70 000	40 000	25	35
F 5	90 000	65 000	22	50
F17	100 000	70 000	20	40
F 6	85 000	55 000	25	60
F22	90 000	65 000	22	50
F 2	70 000	40 000	20	30
F 8	75 000	30 000	45	50
F 8m	75 000	30 000	45	50
F 8c	75 000	30 000	45	50
F 10	80 000	30 000	40	50
F 25	95 000	40 000	35	40

Section 6.—Revise to read as follows:

6. (*a*) ***Bend Tests.***—The bend test specimen specified in Section 7 shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin the diameter of which is equal to the thickness of the specimen.

(*b*) ***Optional Bend Tests.***—If preferred by the manufacturer and approved by the purchaser, the following bend test may be substituted for that described in Paragraph (*a*): A piece of the rolled bar shall stand being bent cold through 90 deg. without cracking on the outside of the bent portion around a pin the diameter of which is equal to the thickness of the section where bent.

Section 8 (*a*).—Revise to read as follows:

8. (*a*) One bend test shall be made from each melt of open-hearth or electric-furnace steel, or from each 25 tons where melts are not identified, or from each blow or lot of 10 tons of bessemer steel.

Section 9.—In Paragraph (*a*) first line, insert the word "tie" before the word "plate."

Revise Paragraph (*b*) to read as follows:

(*b*) When tie plates are specified with rolled crown, a variation of $\frac{1}{32}$ in. in thickness, $\frac{3}{16}$ in. in rolled width and $\frac{1}{4}$ in. in sheared length, $\frac{1}{4}$ in. for the dimensions from the middle of the crown to the sheared edges, and $\frac{1}{4}$ in. for the dimensions from the center of the spike holes to the sheared edges will be permitted.

Add a new Paragraph (*f*) to read as follows:

(*f*) A variation of $\frac{1}{32}$ in. under and $\frac{1}{64}$ in. over in the height of the shoulders will be permitted.

Sections 10, 11, and 12.—Revise to read as follows:

10. ***Weight.***—Tie plates shall be paid for on the basis of actual weight as applied to the entire order, except that payment shall not be made for any weight in excess of 3 per cent over the weight calculated from the specified dimensions.

11. ***Workmanship and Finish.***—The tie plates shall be smoothly rolled and free from injurious warp and other imperfections in surface and projecting fins of metal caused by shearing and punching.

12. ***Marking.***—The tie plate section designation, the name or brand of the manufacturer, and the last two digits of the year of manufacture shall be rolled in raised letters and figures on the outside of the shoulder of the plates, and a portion of this marking shall appear on each finished tie plate.

Standard Specifications for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 46):

Section 3 (*c*).—Revise to read "classes 2H, 3, and 4 nuts shall be made by the hot-forged or cold process, or shall be machined from hot-forged, hot-rolled, or cold-drawn bars and shall be heat treated to meet the required physical properties."

Standard Specifications for Hot-Worked High-Carbon Steel Tie Plates (A 241 – 41):

Section 1.—Revise to read "These specifications cover hot-worked high-carbon steel tie plates for use in railroad track."

Section 3.—Revise the table of chemical requirements to read as follows:

	Open-Hearth or Electric-Furnace
Carbon, per cent	0.35 to 0.82
Phosphorus, max., per cent	0.050
Copper, when specified, min., per cent	0.20

Section 8.—Revise Paragraph (*b*) to read as follows:

(*b*) When tie plates are specified with rolled crown a variation of $\frac{1}{32}$ in. in thickness, $\frac{3}{16}$ in. in rolled width, $\frac{1}{4}$ in. in sheared length, $\frac{1}{4}$ in. for the dimensions from the middle of the crown to the sheared edges, and $\frac{1}{4}$ in. for the dimensions from the center of the spike holes to the sheared edges will be permitted.

Add a new Paragraph (*f*) to read as follows:

(*f*) A variation of $\frac{1}{32}$ in. under and $\frac{1}{64}$ in. over in the height of the shoulders will be permitted.

Sections 9, 10, and 11.—Revise to read as follows:

9. ***Weight.***—Tie plates shall be paid for on the basis of actual weight as applied to the entire order, except that payment shall not be made for any weight in excess of 3 per cent over the

weight calculated from the specified dimensions.

10. *Workmanship and Finish.*—The tie plates shall be smoothly rolled, and free from injurious warp and other imperfections in surface and projecting fins of metal caused by shearing and punching.

11. *Marking.*—The tie plate section designation, the name or brand of the manufacturer, the last two digits of the year of manufacture, shall be rolled in raised letters and figures on the outside of the shoulder of the plates, and a portion of this marking shall appear on each finished tie plate.

EDITORIAL CHANGES IN STANDARDS

Standard Specifications for Steel Helical Springs (A 61 – 39):

Section 6 (a).—Revise to read as follows:

(*a*) *Solid Height.*—The solid height is the perpendicular distance between the plates of the testing machine when the spring is compressed with a test load sufficient to bring all coils in contact, but in no case shall the test load exceed by more than 50 per cent the nominal solid capacity.

Standard Specifications for Steel Elliptical Springs (A 62 – 39):

Sections 7 and 8.—Revise to read as follows:

7. *Compression Method.*—For the compression method of test the load shall be applied gradually and in such a way that the specified working load shall not be exceeded. If it is exceeded, the load shall be released to not more than one-half the specified working load and then gradually increased to the specified working load. The loaded height and the loaded length shall be measured when the spring is in this position. The load shall be increased to one and one-half times the specified working load. The load shall then be fully released and the height measured as free height after the test.

8. *Release Method.*—For the release method of test a load one and one-half times the specified working load shall be applied and then gradually released to the specified working load. If released to less than the specified working load, the load shall again be raised to one and one-half times the specified working load and then gradually released to the specified working load. The loaded height and the loaded length shall be measured when the spring is in this position. The load shall then be fully released and the height measured as free height after test.

Standard Specifications for Heat-Treated Steel Helical Springs (A 125 – 39):

Section 9 (a).—Revise to read as shown above for Section 6 (*a*) in Specifications A 61.

Standard Specifications for Heat-Treated Steel Elliptical Springs (A 147 – 30):

Sections 10 and 11.—Revise to read as shown above for Sections 7 and 8 in Specifications A 62.

REPORT OF COMMITTEE A-3*
ON
CAST IRON

Since the 1947 Annual Meeting of the Society, Committee A-3 on Cast Iron has held one meeting in Washington, D. C. on March 1, 1948. The total membership of the committee is now 127.

At the spring meeting, results were reported of a survey on the use of SR strain gages and similar devices to study stresses in iron castings. The number of users was found sufficient to justify promotion of a symposium on this subject, possibly for the 1949 Annual Meeting.

In Proposed Methods of Impact Testing, it was decided to describe the drop, Izod, and Charpy methods as presently modified for testing cast iron.

New Tentative

The committee recommends for publication as tentative the Proposed Tentative Specifications for Gray Iron Castings for Non-Pressure Containing Parts at Elevated Temperatures, as appended hereto.[1]

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption the following revisions in the Standard Specifications for Gray Iron Castings (A 48 - 46) and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Section 1.—In the last sentence, add the words "and test" after the word "classify."

New Section.—Add a new Section 3 to read as follows, renumbering the subsequent sections accordingly:

3. *Tension Test.*—The tension test shall be considered the primary test for qualification under these specifications. Unless there is agreement between the manufacturer and the purchaser to the contrary, the tension test shall be made and the results of the tension test shall determine compliance with the specifications for the various classes given in Section 2.

Section 3 (a).—Renumber as Section 4 and change to read as follows:

4. (*a*) By agreement between the manufacturer and the purchaser, the tension test may be waived and the transverse test may be specified. When specified, the breaking loads prescribed in Table I shall apply.

Section 3 (d).—Change the first sentence to read as follows: "Where the transverse test has been made by agreement as prescribed in Paragraph (*a*) and the transverse or flexure test bar fails to meet the load requirements prescribed in Table I or the values determined in accordance with Paragraph (*c*), then the manufacturer shall have the right to have tested a tension specimen machined from a broken end of the transverse test bar."

Tentatives Continued Without Revision

The committee recommends that both the Tentative Specifications for Foundry Pig Iron (A 43 - 45 T) and for Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F. (A 278 - 44 T) be continued as tentative. In the case of the latter, additional information on creep strength of various cast irons is considered desirable to facilitate its wider adoption. A survey will be made to determine if sufficient funds can be raised by Committee A-3 to finance such a program. Consultation with the Joint Research Committee on Effect of Temperature on the Properties of Metals was approved.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] These specifications were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Anuual Meeting.[2]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, E. R. Young.
Vice-Chairman, J. S. Vanick.
Secretary, C. O. Burgess.

This report has been submitted to letter ballot of the committee, which consists of 127 members; 80 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. T. MACKENZIE,
Chairman.

C. O. BURGESS,
Secretary.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE A-5*
ON
CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 18, 1947, in Atlantic City, N. J., during the Annual Meeting of the Society, and on March 2, 1948, in Washington, D. C., during the Spring Group Committee Meetings.

Since the last report, 5 new members have been elected and 4 members have resigned; the total membership of the committee is now 130, of whom 59 are classified as producers, 41 as consumers, and 30 as general interest members.

The present officers of the committee have been re-elected for the ensuing term of two years.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following standards be revised, as indicated, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (**A 116 - 39**):[1]

Section 2.—Add the following as a note to this section: "NOTE.—See Appendix to these specifications for table covering design numbers for woven wire fencing, extracted from Simplified Practice Recommendation R9-47."

Appendix.—Add the accompanying Table I as an appendix to these specifications.

Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (**A 121- 39**):[1]

Section 2.—Revise the table in this section to read as follows:

Size, Steel Wire Gage	Nom. Diam. of Zinc-Coated Wire in Strand, in.	No. of Barbs	Barb Spacing, in.	Diam. of Barbs, Steel Wire Gage	Shape of Barbs
No. 12½	0.099	2	4	14	round
No. 12½	0.099	4	5	14[a]	half-round
No. 12½	0.099	4	5	14	round
No. 12½	0.099	2	4	12½[a]	flat
No. 14	0.080	2	4	16	round

[a] The gage of the half-round flat barbs is the same as that of the full-round wire section from which the barbs are made.

These recommended changes in Standards A 116 and A 121 will bring both of these specifications into agreement with the Department of Commerce Simplified Practice Recommendation R9-47.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets (**A 93-46**).[1]—Committee A-5 recommends that these specifications be revised as follows in order to bring them into line with present practice and that they be reverted to tentative:

Table II.—In the title of this table, change "ounce" to read "ounces" and change the weight of coating for No. 28 gage sheets in class B from "1.75" to "2.00" oz. per sq. ft.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1946 Book of A.S.T.M. Standards, Part I-A.

Section 6 (a).—Change the first sentence to read as follows: "The weight of coating shall conform to the requirements prescribed in Table I for the ordered coating and the weight of coating may be determined by any of the methods described in Paragraphs (*c*), (*d*), or (*e*)."

TABLE I.—DESIGN NUMBERS FOR WOVEN WIRE FENCING.

Design Numbers	Number of Horizontal Wires	Height, in.	Spacing of Stay Wires, in.	Size, Steel Wire Gage	
				Intermediate Line and Stay Wires	Top and Bottom Wires
		No. 9 FARM			
1155-12-9	11	55	12	9	9
1047-12-9	10	47	12	9	9
939-12-9	9	39	12	9	9
832-12-9	8	32	12	9	9
726-12-9	7	26	12	9	9
949-12-9	9	49	12	9	9
845-12-9	8	45	12	9	9
635-12-9	6	35	12	9	9
1047- 6-9	10	47	6	9	9
726- 6-9	7	26	6	9	9
		No. 11. FARM			
1047-12-11	10	47	12	11	9
939-12-11	9	39	12	11	9
832-12-11	8	32	12	11	9
726-12-11	7	26	12	11	9
845-12-11	8	45	12	11	9
635-12-11	6	35	12	11	9
1047- 6-11	10	47	6	11	9
939- 6-11	9	39	6	11	9
832- 6-11	8	32	6	11	9
726- 6-11	7	26	6	11	9
		No. 12½ FARM			
1047-12-12½	10	47	12	12½	10
939-12-12½	9	39	12	12½	10
832-12-12½	8	32	12	12½	10
726-12-12½	7	26	12	12½	10
845-12-12½	8	45	12	12½	10
635-12-12½	6	35	12	12½	10
1047- 6-12½	10	47	6	12½	10
939- 6-12½	9	39	6	12½	10
832- 6-12½	8	32	6	12½	10
726- 6-12½	7	26	6	12½	10
		No. 14½ FARM			
939- 6-14½	9	39	6	14½	11
832- 6-14½	8	32	6	14½	11
726- 6-14½	7	26	6	14½	11

NOTE.—The above styles of farm fence are in accordance with the Recommendations of the United States Department of Commerce in its Simplified Practice Recommendation R9–47.

Section 9.—Change Paragraphs (*a*) and (*b*) to read as follows by the addition of the italicized words and numbers and the omission of those in brackets:

9. (*a*) For the triple-spot test three specimens 2.250 ± [0.005] *0.010* in. square or 2.540 ± [0.005] *0.010* in. in diameter shall be cut from each test sheet, one being cut from the center and the others from diagonally opposite corners adjacent to, and within the boundary formed by lines *not less than* 2 in. from the sides and 4 in. from the ends.

(*b*) The bend test specimens, 3 to 4 in. in width, shall be cut from the test sheet within the boundary formed by lines *not less than* 2 in. from the sides and 4 in. from the ends.

Section 10 (a).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

10. (*a*) [The sheet test] *When the sheet weight test method is used it* shall be made on as nearly 1 per cent as practicable, of the sheets of each gage and ordered coating, *or* at approximately hourly intervals.

Section 12 (a).—Change the table covering variations in width to read as follows:

Ordered Width, in.	Permissible Variations Over Ordered Width, in. (to the nearest $\frac{1}{16}$ in.)
Up to 20, incl.	$\frac{1}{8}$
Over 20 to 30, incl.	$\frac{3}{16}$
Over 30 to 50, incl.	$\frac{1}{4}$
Over 50 to 80, incl.	$\frac{5}{16}$
Over 80	$\frac{3}{8}$

Table V.—Change to read as follows:

Ordered Galvanized Sheet Gage	Permissible Variations from Specified Gage Weight[a], per cent Over or Under			
	20 tons and over	Under 20 tons to 3 tons, incl.	Under 3 tons to 1 ton, incl.	Under 1 ton
No. 18 gage and heavier	3.5	5	7.5	10
No. 19 gage and lighter	2.5	3	5	10

[a] The tonnage ordered for shipment to one place at one time determines the permissible variation for that tonnage, although any partial shipment may have the greater permissible variation of its tonnage classification.

Section 12 (b).—Change the present table covering variations in length to read as follows:

Ordered Length, in.	Permissible Variations Over Ordered Length, in. (to the nearest $\frac{1}{8}$ in.)
Up to 15, incl.	$\frac{1}{8}$
Over 15 to 30, incl.	$\frac{1}{4}$
Over 30 to 60, incl.	$\frac{1}{2}$
Over 60 to 96, incl.	$\frac{3}{4}$
Over 96 to 120, incl.	1
Over 120 to 156, incl.	$1\frac{1}{4}$
Over 156 to 192, incl.	$1\frac{1}{2}$
Over 192 to 240, incl.	$1\frac{3}{4}$
Over 240	2

Section 12 (c).—In line three, change "4" to read "6" in.

Section 12 (d).—Change the table on camber deviation to read as follows:

Ordered Length, in.	Camber, in. (to the nearest $\frac{1}{16}$ in.)
Up to 48, incl.	$\frac{1}{8}$
Over 48 to 72, incl.	$\frac{3}{16}$
Over 72 to 96, incl.	$\frac{1}{4}$
Over 96 to 120, incl.	$\frac{5}{16}$
Over 120 to 144, incl.	$\frac{3}{8}$
Over 144 to 168, incl.	$\frac{1}{2}$
Over 168 to 192, incl.	$\frac{5}{8}$
Over 192 to 216, incl.	$\frac{3}{4}$
Over 216 to 240, incl.	$\frac{7}{8}$
Over 240 to 360, incl.	$1\frac{1}{4}$
Over 360 to 480, incl.	$1\frac{1}{2}$

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuance of the following tentatives without change:

Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (**A 219 – 45 T**).[1] These methods are under the joint jurisdiction of Committee A-5 and Committee B-8 on Electroplating. Both committees concur in recommending that these methods remain tentative. Committee B-8 has investigations under way at the present time which it hopes to have completed in time to recommend adopting these methods as standard before the next publication of the Book of Standards in 1949.

Tentative Specifications for Lead-Alloy Coating (Hot Dip) on Iron or Steel Hardware (**A 267 – 46 T**).[1] Studies are under way on the weights of lead coatings which can be applied and it is expected that a report will be made available in a few months. It is intended to incorporate the results of these studies in the specifications.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

RELOCATION OF KEY WEST TEST SITE

The test site at Key West, Fla., was relocated during the early part of 1948 due to the development of this site into a bathing beach. The new site is located at the U. S. Navy Submarine Base.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (W. E. Buck, chairman) has under consideration a proposed revision in the Standard Methods of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A 90 – 39) covering an additional stripping method in which hydrochloric acid diluted with an equal amount of water is used which, it is hoped, will be rapid enough for a shop floor check during production. The Section on Preece Test will review Sections 8 and 12 of the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 – 41) to eliminate the ambiguity in the definition of "end point" as given in these sections.

Subcommittee XI on Sheet Specifications (E. F. Lundeen, chairman) has been occupied with the revisions of Standard A 93, referred to earlier in this report.

Subcommittee XII on Wire Specifications (P. E. Chisler, chairman) prepared the proposed revisions of Standard Specifications A 116 and A 121. A section is studying the need for a specification for stock fence such as is covered by the Department of Commerce Simplified Practice Recommendation R9-47. A review is being made of the Standard Specifications for Zinc-Coated Iron or Steel Chain Link Fence Fabric Galvanized After Weaving (A 117-33) to make it conform with present manufacturing practices. Active work is in progress on a new specification for galvanized high-strength steel telephone and telegraph line wire.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) is conducting the studies referred to earlier in this report on the weights of lead coatings which can be applied as given in Tentative Specifications A 267.

Subcommittee XIV on Sheet Tests (E. S. Taylerson, chairman) continued to make its biennial inspections of sheets at those locations where they are still exposed. A detailed report is appended hereto.[3]

Subcommittee XV on Wire Tests (A. P. Jahn, chairman).—The report of this subcommittee comprises the Report of Wire Inspection Committee on Field Tests of Wire and Wire Products as appended hereto.[4]

Subcommittee XVI on Hardware Tests (L. J. Waldron, chairman).—The report of this subcommittee is appended hereto.[5] There has been considerable discussion in this subcommittee and in Committee A-5 as a whole relative to extending the present hardware tests. It is thought that the hardware tests now under way are too limited in scope and it has been suggested that other committees should be asked to participate so that ferrous and non-ferrous base metal, plastics, paints, and metallic coatings can be tested. The Advisory Subcommittee of Committee A-5 will consult with other committees to obtain their views on this matter.

This report has been submitted to letter ballot of the committee, which consists of 130 members; 85 members have returned their ballots, of whom 77 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. R. GALLOWAY,
Chairman.

H. F. HORMANN,
Secretary.

---

[3] See p. 108.
[4] See p. 114.
[5] See p. 119.

REPORT OF SUBCOMMITTEE XIV ON INSPECTION OF BLACK AND GALVANIZED SHEETS

The atmospheric tests of corrugated sheets at Annapolis, Md., Altoona, Pa., Brunot Island, Pittsburgh, Pa., Sandy Hook, N. J., and State College, Pa., were inspected in both April and October of 1946 and 1947.

Table I shows failures of No. 22 gage sheets at Annapolis (31 yr. exposure).

A new form of table for the other locations (Tables II, III, IV, and V, 21 yr.) gives for each sheet the length of time exposed until the following occurred:

(*a*) First base metal rust,

(*b*) One hundred per cent base metal rust,

(*c*) Visible perforation of the sheet from above.

Other pertinent information is shown in the headings or footnotes.

No table has been prepared for Key West. The zinc coatings on the upper surface of the sheets are quite durable but cannot logically be classified in the same manner as at the other sites because these galvanized sheets perforate by corrosion originating from below. The extent and type of corrosion has been well described in former reports.[1] It was found necessary to remove the Key West racks to a new location at the Naval Submarine Base. Note was made for the future use of the Inspection Committee of slight superficial damage due to blasting.

This report has been submitted to letter ballot of Subcommittee XIV which consists of 15 members; 14 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the subcommittee,

E. S. Taylerson,
Chairman.

---

[1] Report of Committee A-5 on Corrosion of Iron and Steel, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 108 (1940); Vol. 44, p. 95 (1944); Vol. 46, p. 156 (1946).

TABLE I.—RECORD OF FAILURES AT ANNAPOLIS OF NO. 22 GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.
Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1947.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z.....	Open-hearth Steel	B	0.032	0.32	0.063	0.042	..	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	....Z
O.....	Open-hearth Steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	....O
B....	Open-hearth Steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	21.5	... B
M....	Open-hearth Steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	..	..	..	13	13	23.4	....M
B.....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	26.0	28.0	28.5	..	..	..	..	..	..	..	9	9	23.4	....B
Z.....	Open-hearth Steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	27.0	27.5	28.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	27.5	....Z
T.....	Open-hearth Steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	..	..	14	14	22.3	....T
K.....	Open-hearth Steel (Acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	24.5	28.0	28.5	28.5	28.5	29.0	29.5	30.0	31.0	*	*	*	*	*			14	9	..	....K
H.....	Open-hearth Steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	25.0	..	..	..	..	..	..	10	10	22.9	....H
Z.....	Open-hearth Steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	24.5	28.0	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	2	26.3	....Z
Z.....	Open-hearth Steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	28.0	29.0	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	2	..	....Z
S......	Open-hearth Iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	.....S
D....	Open-hearth Iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	..	..	..	..	..	..	..	..	..	..	6	6	18.4	....D
X....	Open-hearth Iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C....	Open-hearth Iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.5	C
C.....	Open-hearth Iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U.....	Open-hearth Iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5		..	..	13	13	19.3	....U
V.....	Open-hearth Iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	..	..	14	14	20.0	V
A.....	Bessemer Steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	....A
Z......	Bessemer Steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	....Z
Z.....	Bessemer Steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	27.5	27.5	27.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	27.5	....Z
I......	Bessemer Steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	26.0	26.5	26.5	26.5	26.5	27.0	28.0	28.0	29.0	29.5	29.5	31.0	*	*	..	15	13	..	I
Z....	Bessemer Steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	28.0	29.5	31.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	29.5	....Z
Z....	Bessemer Steel	B	0.055	0.38	0.110	0.046	..	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	....Z
Y.....	Wrought Iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.3	....Y
E.....	Wrought Iron	A	0.033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	25.0	..	..	..	..	..	..	10	10	22.3	....E
							Total	......	214															Total....		214	203		

---

* Indicates unfailed sheet.

[a] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

TABLE II.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Altoona, Pa., on April 22, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Un-coated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			
	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	Years To Perfo-ration
R (T)	....	....	....	4.3	12.0	21.0	3.3	9.5	18.5	3.3	5.0	14.5	2.3	4.3	14.5	....	....	....	....	....	....	4.3
(B)				4.5	11.5	21.5	3.3	9.0	16.5	3.3	7.0	17.0	2.3	4.5	16.0							
V (T)	4.3	11.0	*	4.3	9.0	b	3.3	7.0	17.5	2.3	7.0	14.5	2.3	5.2	13.5	1.4	3.3	9.0	....	....	....	4.3(V)
(B)	4.3	7.0	*(U)	4.8	9.0	17.5	4.3	6.8	19.0	2.5	7.0	15.0	2.3	4.5	15.0	2.3	3.3	13.0				4.3(U)
N (T)	....	....	....	4.3	8.5	*	3.3	8.5	20.0	3.3	5.5	17.0	2.3	4.5	15.0	1.4	2.8	13.0	2.0	3.3	7.0	7.5
(B)				3.5	12.0	*	3.5	8.0	*	3.3	5.9	19.0	2.3	4.5	b	1.4	3.3	15.0	2.3	3.3	11.0	
T (T)	4.5	10.5	*	5.5	11.5	*	3.5	7.5	20.0	2.3	5.0	18.5	2.3	5.0	15.0	1.4	2.8	13.5	2.0	2.8	9.0	8.5
(B)	4.8	9.0	*	6.3	11.5	*	3.5	7.0	*	2.5	5.2	17.0	2.3	4.3	17.0	2.3	3.0	15.0	2.3	3.3	11.0	
Y (T)	4.3	11.5	17.5	3.5	11.5	15.5	3.3	8.5	17.5	2.5	5.0	14.0	2.3	3.3	14.0	2.3	3.5	4.3	1.4	3.5	10.0	2.0
(B)	4.5	13.7	16.5	3.3	9.5	17.5	4.3	9.0	19.0	3.3	5.2	17.0	2.5	4.3	15.0	2.3	3.3	8.5	2.3	3.3	b	
S (T)	4.3	12.0	*	4.3	12.0	*	4.3	8.5	20.5	3.0	4.5	19.0	2.3	4.0	17.0	2.0	2.8	13.5	2.0	3.0	7.0	9.0
(B)	4.3	12.5	*	4.8	14.5	*	4.3	8.5	*	3.3	6.3	20.5	3.0	4.3	19.0	2.3	2.8	15.0	2.3	3.0	10.5	
C (T)	....	....	....	4.0	10.5	*	3.3	8.5	*	2.5	6.5	21.5	2.3	4.5	19.0	1.4	3.5	b	....	....	....	b
(B)				5.8	13.5	*	4.3	10.0	*	3.3	6.5	19.0	2.5	4.3	19.5	1.4	3.0	b				
K (T)	4.3	12.5	*	4.3	11.5	*	4.3	7.5	*	3.3	5.2	21.5	2.5	4.3	b	2.3	3.0	b	2.3	2.8	10.5	13.0
(B)	4.3	11.5	*	4.5	11.5	*	4.3	8.5	*	3.3	5.0	*	3.3	4.3	21.0	2.3	3.0	21.0	2.3	3.0	10.5	
W (T)	....	....	....	5.2	10.5	21.5	3.5	6.5	19.0	3.3	5.0	15.0	2.3	4.0	14.5	....	....	....	....	....	....	5.0
(B)				5.2	12.0	*	4.3	7.5	19.5	3.3	5.5	17.0	2.3	4.0	15.0							
Aver-age	4.4	11.1		4.6	11.2		3.8	8.1		3.0	5.7		2.4	4.3		1.9	3.1		2.1	3.1		

* Data not yet available.
[a] Coating weight is of sheet, and is the sum of the weights on each side.
[b] Blown away or discarded due to mechanical damage.

TABLE III.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Brunot Island, Pittsburgh, Pa., on April 21, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Un-coated
	Years to			Years To			Years To			Years To			Years To			Years To			Years To			
	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	Years To Perforation
R (T)	5.3	13.5	*	5.3	13.0	19.5	4.3	14.4	20.0	3.5	8.5	17.5	3.3	6.7	15.5	....	....	....	....	....	....	2.0
(B)	5.5	14.4	*	5.8	13.5	*	4.5	13.0	18.5	3.8	9.5	17.5	3.3	6.7	17.0							
V (T)	5.5	13.5	*	4.0	12.5	19.5	4.3	9.5	*	3.3	11.3	18.5	3.3	7.5	17.5	2.0	4.5	15.5	....	....	....	2.3(V)
(B)	5.0	12.0	*(U)	5.5	13.0	*	4.8	10.5	20.6	3.3	10.5	18.5	3.3	6.7	18.5	2.3	4.5	13.5				2.5(U)
N (T)	....	....	....	5.3	12.5	*	4.5	11.5	*	3.3	9.5	19.5	2.5	5.5	19.0	2.0	3.5	16.5	2.3	3.5	10.5	3.0
(B)				5.3	15.5	*	4.8	11.5	*	3.8	8.5	*	3.3	6.0	19.5	2.0	3.5	18.0	2.3	4.5	10.5	
T (T)	5.3	12.5	*	6.3	13.5	*	4.0	11.0	*	3.3	6.7	*	2.5	6.5	*	2.3	4.3	19.0	2.0	3.0	10.5	9.0
(B)	5.8	13.5	*	8.5	15.5	*	4.5	11.0	*	3.3	8.5	*	3.3	6.0	*	2.3	4.5	19.5	2.3	4.3	12.5	
Y (T)	4.8	14.4	11.5	4.5	11.5	19.0	3.5	11.0	17.0	3.5	6.7	15.5	3.3	4.8	15.5	2.0	4.3	20.0[b]	2.0	3.8	19.0[b]	2.0
(B)	5.5	16.5	11.5	5.5	14.0	20.6	4.5	16.5	14.0	3.3	8.3	17.5	3.3	6.0	13.0	2.3	5.0	20.0	2.3	4.5	[b]	
S (T)	4.3	15.5	*	5.3	15.0	*	4.5	10.5	*	3.5	7.4	19.0	3.3	5.5	21.5	2.3	4.8	14.5	2.3	3.3	4.0	3.3
(B)	5.3	15.5	*	7.0	18.5	*	5.3	12.5	*	3.8	7.4	*	3.3	6.0	*	2.3	3.5	18.0	2.3	4.0	4.3	
C (T)	....	....	....	4.3	12.0	*	4.0	11.5	*	3.3	10.5	*	3.3	6.3	*	2.0	4.3	*	....	....	....	12.5
(B)				8.5	18.5	*	4.8	11.0	*	3.3	10.5	*	3.3	6.8	*	2.3	4.5	*				
K (T)	4.8	14.0	*	5.5	15.0	*	4.3	10.5	*	4.0	8.3	*	3.3	5.0	*	2.3	3.5	*	2.3	3.0	14.5	16.5
(B)	5.3	15.5	*	7.3	16.5	*	5.8	12.5	*	4.3	8.5	*	3.3	6.5	*	2.3	4.0	*	2.3	3.5	14.5	
W (T)	....	....	....	5.5	12.5	*	4.5	9.5	21.5	3.3	7.3	18.0	3.3	5.5	17.5	....	....	....	....	....	....	3.0
B)				6.3	15.5	*	5.8	12.5	*	3.3	7.3	13.0	3.3	6.0	19.5							
Average	5.2	14.3		5.9	14.4		4.6	11.7		3.5	8.6		3.2	6.1		2.2	4.2		2.2	3.7		

* Data not yet available.

[a] Coating weight is of sheet and is the sum of the weights on each side.

[b] Blown away or discarded due to mechanical damage.

TABLE IV.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Sandy Hook, N. J., on May 20, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Un-coated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			
	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	Years To Perforation
R (T)	13.4	*	*	12.4	*	*	8.4	*	*	6.4	17.9	*	7.4	14.4	*	....	....	....	....	....	....	5.9
(B)	13.4	*	*	12.9	*	*	9.4	*	*	7.4	19.4	*	6.9	15.9	*							
V (T)	12.4	*	*	10.9	*	*	9.9	19.4	*	7.4	19.9	*	6.4	19.4	*	5.4	13.4	*	....	....	....	4.9(V)
(B)	10.9	*	*(U)	12.4	*	*	10.4	19.4	*	6.9	20.4	*	7.4	15.4	*	4.5	11.0	*				5.4(U)
N (T)	....	....	....	11.4	*	*	9.4	*	*	7.4	18.4	*	6.9	15.4	*	4.5	11.4	*	4.5	11.4	*	5.9
(B)				10.4	*	*	9.9	20.9	*	7.4	17.4	*	5.9	15.4	*	4.5	11.0	*	6.4	12.0	*	
T (T)	12.4	*	*	15.9	*	*	9.9	*	*	6.9	17.9	*	5.9	15.9	*	4.5	13.4	*	4.5	13.4	20.9	6.4
(B)	12.4	*	*	16.4	*	*	9.9	19.4	*	6.9	14.4	*	5.4	15.4	*	4.5	11.0	*	5.4	12.0	*	
Y (T)	10.9	*	*	11.4	*	*	8.4	*	*	7.4	17.4	*	6.4	16.4	*	4.5	11.0	*	4.5	12.0	*	3.4
(B)	12.4	*	*	13.4	*	*	8.4	*	*	8.4	17.9	*	6.9	13.4	*	5.4	13.4	*	4.5	11.4	*	
S (T)	10.4	*	*	11.9	*	*	9.9	21.4	*	7.4	15.9	*	5.9	12.9	*	4.5	9.9	*	4.5	9.4	20.9	9.9
(B)	11.4	*	*	13.4	*	*	12.4	*	*	9.4	16.9	*	7.4	15.4	*	4.9	9.4	*	5.4	9.4	*	
C (T)	....	....	....	11.4	*	*	8.4	*	*	7.4	19.4	*	6.9	15.4	*	4.5	12.4	*	....	....	....	10.4
(B)				18.4	*	*	9.4	*	*	7.4	19.4	*	7.4	14.4	*	4.5	11.4	*				
K (T)	10.9	*	*	11.4	*	*	10.4	20.9	*	8.4	15.9	*	7.4	13.9	*	4.5	9.4	*	4.5	10.0	*	12.4
(B)	10.4	*	*	12.9	*	*	11.4	*	*	8.4	17.4	*	7.4	13.9	*	6.4	10.4	*	4.9	10.0	*	
W (T)	....	....	....	13.4	*	*	11.4	*	*	8.4	17.4	*	7.4	14.4	*	....	....	....	....	....	....	4.9
(B)				14.4	*	*	11.4	*	*	7.4	19.4	*	6.4	16.4	*							
Average	11.8			13.1			9.9			7.6	17.9		6.8	15.2		4.8	11.3		4.9	11.1		

* Data not yet available.

[a] Coating weight is of sheet and is the sum of the weights on each side.

TABLE V.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at State College, Pa., on April 23, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Un-coated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			Years To Perfo-ration
	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	First Rust	100 per cent Rust	Perforation	
R (T)	*	*	*	*	*	*	19.0	*	*	17.5	*	*	14.5	*	*	....	....	....	....	....	....	*
(B)	*	*	*	*	*	*	20.0	*	*	16.5	*	*	15.0	*	*							
V (T)	*	*	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	*	*	....	....	....	*(V)
(B)	*	*	*(U)	*	*	*	*	*	*	15.5	*	*	16.5	*	*	10.5	*	*				*(U)
N (T)	....	....	....	20.5	*	*	*	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	*	*	*
(B)				*	*	*	*	*	*	15.5	*	*	13.5	*	*	9.0	*	*	11.5	*	*	
T (T)	*	*	*	*	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	*	*	9.0	*	*	*
(B)	*	*	*	*	*	*	*	*	*	14.5	*	*	13.5	*	*	10.5	*	*	12.0	*	*	
Y (T)	21.5	*	*	*	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	*	*	9.5	*	*	*
(B)	*	*	*	*	*	*	19.0	*	*	18.5	*	*	14.5	*	*	10.5	*	*	13.0	*	*	
S (T)	*	*	*	*	*	*	*	*	*	19.0	*	*	14.5	*	*	10.5	21.5	*	11.5	*	*	*
(B)	*	*	*	*	*	*	*	*	*	19.5	*	*	15.5	*	*	11.0	21.5	*	11.5	21.5	*	
C (T)	....	....	....	*	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	*	*	....	....	....	*
(B)				*	*	*	21.5	*	*	18.5	*	*	14.5	*	*	10.0	*	*				
K (T)	*	*	*	*	*	*	*	*	*	17.5	*	*	14.5	*	*	11.5	*	*	10.5	*	*	*
(B)	*	*	*	*	*	*	*	*	*	21.0	*	*	16.5	*	*	11.5	*	*	11.5	21.0	*	
W (T)	....	....	....	*	*	*	*	*	*	16.5	*	*	13.5	*	*	....	....	....	....	....	....	*
(B)				*	*	*	*	*	*	15.0	*	*	14.5	*	*							
Aver-age										17.0			14.6			10.0			11.1			

* Data not yet available.

[a] Coating weight is of sheet and is the sum of the weights on each side.

REPORT OF SUBCOMMITTEE XV ON FIELD TESTS OF WIRE AND WIRE PRODUCTS

(COVERS 1947 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1947. Detailed results are reported in the odd-numbered years, and reference to these earlier reports of Committee A-5 on Corrosion of Iron and Steel, which appeared in the Society's *Proceedings* for 1939, 1941, 1943, 1945, and 1947, will be helpful in studies of these exposure test results.

The materials under test are mostly bare steel and zinc-coated wires and wire products, but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight in terms of ounces per square foot of surface. For ready comparisons of coating thickness, 1 oz. per sq. ft. of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0010 in. thick for[1] lead coatings and 0.0013 in. thick for copper coatings. Specimens of wires and wire products have been exposed for about eleven years at eleven locations. These locations have severely industrial, mildly industrial, rural or seacoast atmospheres. The location of these sites and their general classification regarding atmospheres are as follows:

Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa.	Severe Industrial	On Brunot Island in the Ohio River about 2 miles west of the city.
Bridgeport, Conn.	Industrial	In the city
Sandy Hook, N. J.	Seacoast	About 300 yd. from Atlantic Ocean
Santa Cruz, Calif.	Seacoast	About 3 miles from Pacific Ocean
State College, Pa.	Rural	...
Lafayette, Ind.	Rural	...
Ames, Iowa	Rural	...
Manhattan, Kans.	Rural	...
Ithaca, N. Y.	Rural	...
College Station, Texas	Rural	..
Davis, Calif.	Rural	About 80 miles inland

The extent of corrosion is being measured by visual examination of all specimens at the test site and by tension tests on the unfabricated wire specimens. Loss of weight tests on lengths of zinc-coated wire were completed earlier in this corrosion study and the results were reported in the above mentioned *Proceedings*. During 1947, the Pittsburgh specimens were inspected in the spring; Bridgeport, Sandy Hook, and State College specimens were inspected in the spring and fall by a traveling committee. Inspections were made in the fall at the other test sites by the university people in charge of the test plots.

Summaries of the records of inspection through 1947 are shown in the condensed Tables A, B, C, D, E, and F.

Up to the present time (11 years of exposure) no marked deterioration of the

---

[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analysis of lead coatings.

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion o better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green Br = brown; PHR and PHY;= rust or yellowed in pinholes.

Location	Coating Group, oz. per sq. ft. of surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.85	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
Sandy Hook, N. J.	100R	100R	100R	100R	100R	100R	100R	99R	75R	6R	G
Bridgeport, Conn.	100R	100R	100R	100R	100R	100R	100R	100R	90R	6R	GY
State College, Pa.	100R	100R	100R	99R	60R	35R	14R	1R	GY	G	G
Lafayette, Ind.	100R	100R	100R	100R	95R	75R	50R	25R	G	MG	MG
Ames, Iowa	100R	95R	90R	10R	1R	2R	1R	G	G	G	G
Manhattan, Kans.	35R	30R	7R	5R	1R	3R	1R	1R	MG	MG	MG
Ithaca, N. Y.	100R	100R	100R	99R	60R	30R	20R	1R	GY	M	M
Santa Cruz, Calif.	GY	GY	GY	GY	G	G	G	G	...	MG	MG
College Station, Tex.	50R	10R	1R	2R	1R	2R	1R	1R	G	G	G
Davis, Calif.	2R	1R	G	G	G	G	G	G	...	MG	MG

TABLE B.—CORROSION CONDITIONS OF UNFABRICATED WIRES CARRYING COATINGS OTHER THAN ZINC AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pinholes.

Location	Coating Groups and Corrosion Resistant Groups				
	Copper-Covered Wires[a]	Lead-Coated Wires[b]	12 to 14% Chromium, Cold Drawn	12 to 14% Chromium, Air Quenched	18% Chromium, 8% Nickel
Pittsburgh, Pa.	D	D70R	DSR	DSR	D
Sandy Hook, N. J.	Gn	100R+Y	YSR	YSR	GY
Bridgeport, Conn.	Gn	3R+Y	Y	GY	MG
State College, Pa.	Gn	100R+Y	MG	MG	M
Lafayette, Ind.	Gn	60PHR	M	M	M
Ames, Iowa	Br	20R	SY	SY	MY
Manhattan, Kans.	Br	15PHY	SR	G	M
Ithaca, N. Y.	Br	5R+Y	SY	SY	M
Santa Cruz, Calif.	Br	20SR	65SR	25SR	MG
College Station, Tex.	Br	20R+Y	95R+Y	80R+Y	M
Davis, Calif.	Br	30SR	45SR	15SR	MG

[a] Weight of coating 7 to 9 oz. per square foot of surface.
[b] Weight of coating 1.4 to 1.9 oz. per square foot of surface.

TABLE C.—CORROSION CONDITIONS OF FARM FIELD FENCE AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. zinc per sq. ft. of surface							Copper-Covered[a]	Lead-Coated[b]	Chromium-Nickel Steel
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 or more			
State College, Pa.	100R	100R	99R	65R	50R	20R	GY	Gn	100R+Y	MG
Lafayette, Ind.	100R	95R	90R	90R	75R	40R	5R	Gn	40PHR	M
Ames, Iowa	100R	60R	50R	10R	8R	G	G	Br	20R	SY
Manhattan, Kans.	15R	10R	GY	GY	G	MG	MG	Br	10R	MG
Ithaca, N. Y.	100R	90R	80R	40R	25R	5R	1R	Gn	10R	MG
Santa Cruz, Calif.	3R	10R	GY	GY	G	G	MG	Br	30SR	50R
College Station, Tex.	30R	20R	3R	2R	GY	2R	G	Br	7R	SR
Davis, Calif.	3R	5R	G	G	G	MG	MG	Br	40SR	10SR

[a] Weight of coating 7.42 oz. per sq. ft. of surface.
[b] Weight of coating 1.48 oz. per sq. ft. of surface.

TABLE D.—CORROSION CONDITIONS OF BARBED WIRE AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. zinc per sq. ft. of surface							Copper-Covered[a]	Lead-Coated[b]
	0.20 to 0.30	0.35 to 0.45	0.50 to 0.60	0.70 to 0.85	0.80 to 1.00	1.60 to 1.80	2.00 or more		
State College, Pa.	100R	100R	40R	G	GY	G	G	Gn	...
Lafayette, Ind.	100R	100R	70R	10R	2R	1R	1R	Gn	30PHR
Ames, Iowa	100R	60R	3R	G	G	G	G	Br	5R
Manhattan, Kans.	35R	GY	G	MG	MG	MG	MG	Br	10R
Ithaca, N. Y.	100R	95R	15R	1R	G	MG	M	Gn	5R
Santa Cruz, Calif.	1R	GY	GY	MG	G	G	MG	Br	25SR
College Station, Tex.	10R	2R	1R	G	G	G	G	Br	25R+Y
Davis, Calif.	1R	G	G	MG	MG	MG	MG	Br	25SR

[a] Weight of coating 3.97 oz. per sq. ft. of surface.
[b] Weight of coating 1.83 oz. per sq. ft. of surface.

TABLE E.—CORROSION CONDITIONS OF CHAIN LINK FENCE AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. zinc per sq. ft. of surface					Copper-Covered[c]	Lead-Coated[d]	Corrosion Resistant
	0.29 to 0.52[a]	1.13 to 1.22[a]	2.09 to 2.92[a]	1.40 to 1.97[b]	4.21[b] or more			
Pittsburgh, Pa.	100R	100R	100R	100R	80R	...	50R	D
Sandy Hook, N. J.	100R	100R	G	50R	GY	Gn	60R+Y	M
Bridgeport, Conn.	100R	25R	G	45R	GY	Gn	Y	MG
State College, Pa.	90R	GY	G	G	GY	Gn	100R+Y	M
Lafayette, Ind.	99R	G	MG	10R	MG	Gn	50PHR	M
Ithaca, N. Y.	90R	G	MG	G	M	Br	5R	M
Santa Cruz, Calif.	GY	MG	MG	MG	MG	Br	25SR	MG
Davis, Calif.	3R	MG	MG	MG	MG	Br	35SR	MG

[a] Coated before weaving.
[b] Coated after weaving.
[c] Weight of coating 8.52 oz. per sq. ft. of surface.
[d] Weight of coating 1.75 oz. per sq. ft. of surface.

TABLE F.—CORROSION CONDITIONS OF WIRE STRAND AFTER APPROXIMATELY ELEVEN YEARS EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. zinc per sq. ft. of surface				Lead-Coated[a]
	0.69 to 0.93	1.03 to 1.17	1.32 to 1.51	1.71 to 2.47	
Pittsburgh, Pa.	100R	100R	90R	50R	50R
Sandy Hook, N. J.	20R	3R	GY	G	100R+Y
Bridgeport, Conn.	20R	2R	GY	GY	95R+Y
State College, Pa.	G	G	G	G	90R+Y
Lafayette, Ind.	MG	G	MG	MG	50PHR
Ames, Iowa	G	G	G	G	17R
Manhattan, Kans.	MG	MG	MG	MG	50PHY
Ithaca, N. Y.	MG	MG	MG	M	5R
Santa Cruz, Calif.	G	G	G	MG	45SR
College Station, Tex.	G	G	G	G	90R+Y
Davis, Calif.	G	MG	MG	MG	50SR

[a] Weight of coating 1.96 oz. per sq. ft. of surface.

corrosion-resistant steel wires has been noted. In general, at all locations the 18 per cent chromium—8 per cent nickel wires retained their metallic luster. The 12 to 14 per cent chromium types have shown some discoloration, yellowing, or rust spotting of a superficial nature. The cold-drawn wires have darkened more than the air-quenched wires.

A close inspection of the wires exposed at Bridgeport, Sandy Hook, and Pittsburgh was made with a 14× magnifying glass. At Bridgeport the air-quenched wires still have a fairly bright appearance. Several very small pits could be detected haphazardly scattered over the entire surface. The surface of the cold-drawn wires is somewhat darker in color. There is no evidence of red rust. The pits in this type of wire are also very small and non-uniformly distributed and seem to be slightly larger and deeper than those found on the air-quenched specimens.

The wires exposed at Sandy Hook, under a 14× magnifier show pitting in all cases somewhat more pronounced than at Bridgeport. There is a definite directional effect noticeable in that the surfaces of the cold-drawn wires facing the east (ocean side) have a considerable quantity of red rust spotting. The surfaces facing west (inland) are practically free from red rusting. The air-quenched wires here, too, are less dark than the cold-drawn specimens, although not as bright as at Bridgeport. Besides the rust spots noticed above, the cold-drawn wires are covered with a tightly adherent coating of an undetermined nature.

A close scrutiny of the Pittsburgh specimens with a 14 power glass revealed that all the cold-drawn wires were coated with a heavy, rough and tightly adherent scale, very dark gray in color. There is some rusting noticeable, which, however, is generally not too pronounced. Pitting has occurred in all three sizes of cold-drawn wire, and here, as in the other two locations, it definitely becomes worse as the diameter of the wire decreases.

The air-quenched wires in most cases appear to have a thinner scale than the cold-drawn specimens. Pitting is more pronounced than at either of the other two test locations. Because of the relatively thinner coating, a considerable amount of pitting is visible even on the thicker wires (0.135-in. diameter). In contrast to the marked difference between cold-drawn and air-quenched wires at Bridgeport and at Sandy Hook—regardless of size—the No. 18-gage (0.047-in. diameter) air-quenched wire at Pittsburgh is as severely corroded and pitted as the No. 17-gage (0.053-in. diameter) cold-drawn wire. No directional effect, such as was noted at Sandy Hook, was evident in this case. It was the opinion of the inspection group that none of the wires had deteriorated sufficiently to warrant making tensile strength tests at this time.

During 1947, tensile strength test specimens were taken from all the sites except State College. From Bridgeport, 7 bare steel wires and 20 zinc-coated wires were taken; from Sandy Hook, 18 zinc-coated wires were removed; and from Pittsburgh, 4 bare steel wires and 10 zinc-coated wires were taken. None of these specimens showed any significant deviation from the previously reported rate of tensile strength loss.

One set of four lead-coated wires was removed from the Pittsburgh site for tensile strength tests. Previous tests, made after $1\frac{1}{2}$, $3\frac{1}{2}$, and $6\frac{1}{2}$ yr. of exposure had shown no significant decrease in tensile strength of these lead-coated wires. The 1947 test ($10\frac{1}{2}$ yr. of exposure) showed a tensile strength loss of 9 per cent for wire No. 040, 11 per cent for wire No. 041, 9 per cent for wire No. 042, and 21 per cent for wire No. 043. The No. 043 wire is the smallest lead-coated wire of the lot.

From each of the remaining seven university test plots one set of 19 bare steel wires was taken for tensile strength tests. Previous removals from these sites were made seven years ago. At that time strength losses were so low that they could not be considered significant. In Table G are shown the results of the recent tests which in most cases indicate an appreciable loss in strength, particularly of the smaller wires. On the basis of tests to date the loss in tensile strength by uncoated steel wires expressed in per cent per year of exposure for the various gages of wires is as shown in Table H. Although these data are for uncoated wire, other data have indicated that after the coating has gone, the rate of loss is the same whether wires were previously coated or not.

TABLE G.—PERCENTAGE LOSS OF TENSILE STRENGTH AFTER ELEVEN YEARS EXPOSURE.

Wire Lot[b]	Lafayette, Ind.	Santa Cruz, Calif.	Ithaca, N. Y.	Ames, Iowa	College Sta., Tex.	Davis, Calif.	Manhattan, Kan.
No. 501[a]	40.0	35.0	28.0	26.0	24.0	15.0	7.0
No. 506	42.3	32.7	35.0	30.8	24.0	19.2	5.8
No. 507	42.0	40.4	34.5	31.0	26.1	18.5	3.4
No. 400[a]	21.9	19.7	14.6	14.6	9.4	4.2	1.0
No. 401[a]	28.9	17.8	18.9	18.9	14.4	7.8	1.0
No. 405	25.8	18.5	19.6	21.7	13.0	10.3	0
No. 406	23.4	14.9	14.8	13.8	8.5	7.5	0
No. 300[a]	15.5	10.1	7.5	10.8	7.4	4.7	0
No. 302[a]	25.0	17.3	16.8	16.8	19.1	13.2	5.9
No. 305	22.1	13.8	12.9	15.2	12.0	7.4	1.4
No. 306	15.5	13.4	7.0	10.5	8.5	4.9	0
No. 200[a]	19.3	13.9	12.0	17.3	11.5	9.1	2.4
No. 202[a]	...	7.9	7.3	...	6.1	9.5	...
No. 205	14.0	9.5	9.6	5.9	3.2	6.3	1.0
No. 206	17.9	10.8	11.5	12.5	8.5	8.8	3.4
No. 101[a]	10.6	4.8	5.6	7.1	3.2	5.4	1.1
No. 102	13.8	10.7	10.0	9.9	8.6	9.1	5.5
No. 105[a]	12.7	8.2	7.4	9.4	2.0	2.7	0
No. 106	9.2	5.4	6.5	7.1	4.8	3.7	0

[a] Low copper content wires 0.05 to 0.08 per cent copper. All other wires have approximately 0.25 per cent copper.

[b] 500 series—No. 14.5 gage wires. 400 series—No. 12.5 gage wires. 300 series—No. 11 gage wires. 200 series—No. 9 gage wires. 100 series—No. 6 gage wires.

At Manhattan, Kans., F. C. Fenton has broadened the exposure test to the extent of exposing additional farm field fence specimens to specialized conditions. Soil blows off the fields and piles up in the fence rows where it is stopped by the grass and the fence, thus covering the lower portion of the fences with drifted soil. Specimens of twelve farm fences were erected in October 1947, set low so that the bottom three line wires are covered with soil. These specimens will be examined periodically to determine the effect of the soil coverage on rate of corrosion of zinc-coated wires. Another set of twelve farm field specimens was exposed in October, 1947 to study the effect of annual burning of weeds and stubble along fence rows on the rate of corrosion of zinc-coated wires. The specimens erected were:

TABLE H.—LOSS OF TENSILE STRENGTH—UNCOATED STEEL WIRES.

Exposure Site	Loss in Tensile Strength, per cent per yr. exposure				
	No. 14½ Gage	No. 12½ Gage	No. 11 Gage	No. 9 Gage	No. 6 Gage
Pittsburgh, Pa.	14	10	7	6	4
Sandy Hook, N. J.	10	6	4	3	2
Bridgeport, Conn.	6	3	2	2	1
State College, Pa.	5	2	2	2	1
Lafayette, Ind.	4	2	2	2	1
Santa Cruz, Calif.	3	2	1	1	
Ithaca, N. Y.	3	2	1	1	
Ames, Iowa	3	2	1	1	
College Station, Tex.	2	1	1		
Davis, Calif.	2	1			
Manhattan, Kans.	1				

For Burning Tests		For Soil Coverage Tests	
Specimen Number	Specimen Number	Specimen Number	Specimen Number
A410	A338	A412	A236
A215	A246	A216	A247
A315	A348	A317	A347
A225	A248	A227	A252
A325	A350	A327	A352
A337	A397	A336	A298

This report has been submitted to letter ballot of Subcommittee XV which consists of 24 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the subcommittee,

A. P. JAHN,
Chairman.

REPORT OF SUBCOMMITTEE XVI ON FIELD TESTS AND INSPECTIONS OF BARE AND METALLIC COATED HARDWARE, STRUCTURAL SHAPES, PLATES, BARS, AND THEIR PRODUCTS

The data obtained in the routine semiannual inspection of the three remaining test locations have been assembled in Table I. These cover the period from April, 1946, to December, 1947.

A brief résumé of the status of the tests at each location follows:

Altoona and Brunot Island, Pa.:

All tests at these locations have been discontinued. Last tabulation was in the 1944 report.[1]

State College, Pa.:

Only zinc-, aluminum-, and lead-coated specimens remain. In general, no great change has occurred in the appearance of any of the specimens since the 1946 Report[2]. Hot-dip zinc-coated specimens show some alloy rusting with no appreciable appearance of deep red rust from the basis metal. Aluminum-coated specimens are relatively free of rust except at threaded areas. The surface of the lead-coated specimens is almost entirely covered with pinhole rust with a small amount (5 to 10 per cent) of deep-seated scale characteristic of rusting of the basis metal.

Sandy Hook, N. J.:

Only zinc-, aluminum- and lead-coated specimens remain on exposure. Most of the hot-dip galvanized specimens show small amounts of yellow—with slight amounts of red rust. The hot-dip aluminum-coated specimens were relatively free of rust but darkened somewhat; the appearance and disappearance of the yellow type of pinhole rusting, previously noticed on the aluminum-coated samples, was again observed. All lead-coated specimens show a high percentage of pinhole rusting accompanied by 5 to 10 per cent of rusting of the basis metal.

Key West, Fla.:

Several kinds of specimens were so mixed up on the rack that their identifications were lost. Accordingly these have been omitted from this report. The following summary is based upon the 1947 inspections:

All Parkerized samples have completely rusted. Complete rusting has also occurred on most of the thin electrogalvanized specimens. Some of the medium or heavily coated electrogalvanized specimens show 0 to 50 per cent rust. Practically all specimens of hot-dip galvanizing are free from any type of rust. Most all of the hot-dip aluminum specimens show a yellow pinhole type of rusting or staining (0 to 30 per cent) accompanied by a characteristic white incrustation of corrosion products. All of the lead-coated specimens are completely covered with pinhole red rust and stain characteristic of these types of coatings. Considerable lead coating still remains as evidenced by scraping tests.

---

[1] Report of Subcommittee VIII on Field Tests of Metallic Coatings, Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 92, (1944).

[2] Report of Subcommittee VIII, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 154 (1946).

TABLE I.—REPORT OF INSPECTIONS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC.; EXPOSED TO ATMOSPHERE ON TEST RACKS AT THREE LOCATIONS.

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; R*, rust spots widely distributed; Y, yellow or orange yellow rust; D, black or dark surface films (soot or dirt); Tr, trace; X, 100 per cent rust listed in previous reports; TH, threads; a, samples mixed; b, average of nipple and ell. Blank spaces indicate samples removed in 1942.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.				Exposure at Sandy Hook, N. J.				Exposure at Key West, Fla.	
		4-17-46 886 Weeks	10-17-46 912 Weeks	4-23-47 938 Weeks	10-16-47 963 Weeks	4-16-46 894 Weeks	10-16-46 920 Weeks	4-22-47 947 Weeks	10-15-47 972 Weeks	4-10-47 951 Weeks	12-29-47 988 Weeks
3 by 3 by 5/16-in. Plain Steel Angles											
Electrogalvanized	0.52									10R	10R
Hot-dip galvanized	2.52	0	0	0	0	Tr-R, Y	5Y	10Y	15Y	0	0
Hot-dip aluminum	0.89	0D	0	0	0	Tr-Y	5Y	10Y	15Y	Tr-Y	Tr-Y
Cadmium-plated	0.09									100R	100R
Electric Sherardized	1.38	100Y	100Y	100Y	100Y					100Y	100Y
Lead coated	...	90R*	95R*	95R*	5R-95R*					100R*	100R*
3 by 3 by 5/16-in. Copper-Bearing Angles											
Electrogalvanized	0.37									15R	10R
Hot-dip galvanized	2.88	5Y	7Y	10Y	5Y	0	0	0	0	0	0
Hot-dip aluminum	0.82	0D	0	0	0	0	0	0	0	0	0
Cadmium-plated	0.24									60R	75R
Electric Sherardized	1.44	1R-99Y	1R-99Y	5R-95Y	1R-99Y					100Y	100Y
Lead coated	...	90R*	5R-95R*	10R-90R*	5R-95R*					100R*	90R*
2 by 1½ by 3/16-in. Plain Steel Angles											
Electrogalvanized	0.53									50R	60R
Electrogalvanized	0.50									10-95R	15-100R
Hot-dip galvanized	3.24	Tr-Y	Tr-Y	Tr-Y	Tr-Y	5Y	Tr-R	3R-5Y	3R-5Y	0	0
Hot-dip aluminum	0.75	0	0	0	0	Tr-R	Tr-R	Tr-R	Tr-R	30R*	50R*
Cadmium-plated	0.43									80R	98R
Electric Sherardized	1.21									20R-80Y	100R
Lead coated	...	Tr-R, 70R*	5R-90R*	5R-90R*	5R-90R*	Tr-R, 40R*	1R-60R*	5R-60R*	1R-60R*	100R*	90R*
2 by 1½ by 3/16-in. Copper-Bearing Angles											
Electrogalvanized	0.70									35R	60R
Electrogalvanized	0.76									7-50R	20-90R
Hot-dip galvanized	2.75	0	Tr-Y	Tr-Y	Tr-Y	5Y	Tr-R	3R-5Y	3R-5Y	0	0
Hot-dip aluminum	0.50	0	0	0	0	Tr-R	Tr-R	Tr-R	Tr-R	10R*	5Y
Cadmium plated	0.50									70R	85R
Electric Sherardized	1.15									100R-X	100R-X
Gas Sherardized	0.17									100R-X	100R-X
Lead coated	...	Tr-R, 80R*	5R-90R*	5R-95R*	5R-90R*					100R*	98R*

2-IN. DIAMETER TUBULAR GOODS											
Electrogalvanized	1.58	0	0	0	0					0	0
Hot-dip galvanized	2.51	Tr-Y	Tr-Y	5Y	Tr-Y	15Y	Tr-Y	10Y	15Y	0	0
Hot-dip aluminum	4.73	0	0	0	0	Tr-R	Tr-Y,Tr-R	0	Tr-Y	0	10Y
Cadmium plated	0.23									70R	80R
Cadmium plated	0.10									100R	100R
Electric Sherardized	0.29									100Y	100Y
Electric Sherardized	0.85									100Y	100Y
Gas Sherardized	0.66	10R-90Y	20R-80Y	30R-70Y	30R-70Y*					100Y	100Y
Lead coated	...	2R-90R*	5R-95R*	5R-95R*	5R-95R*	5R-80R*	10R-90R*	10R-80R*	10R-90R*	100R*	100R*
1-IN. DIAMETER TUBULAR GOODS											
Electrogalvanized	1.62	0	0	0	0					0	0, Tr-Y
Hot-dip galvanized	1.83	90Y	100Y	5R-95Y	100Y					0	0
Hot-dip aluminum	1.83	0	0	0	0					10R*	2Y
Electric Sherardized	0.92	5R-95Y	10R-90Y	15R-85Y	20R-80Y					100Y	100Y
Gas Sherardized	0.72	25R-75Y	40R-60Y	40R-60Y	40R-60Y					100Y	100Y
Calorized	...	10R*-D	100Y-D	90Y-10R*	100Y-D					(a)	(a)
Lead coated	...	1R-90R*	5R-95R*	5R-95R*	5R-95R*					100R*	95R*
Amaloy	...										
FORGED STEEL TOWER FITTINGS											
Hot-dip galvanized	2.67	40Y	30Y	30Y	50Y					(a)	(a)
Hot-dip galvanized	1.53	40Y	30Y	50Y	50Y					(a)	(a)
Hot-dip aluminum	...	0	0	0	0					(a)	(a)
Electric Sherardized	0.50	20R-80Y	25R-75Y	25R-75Y	25R-75Y					(a)	(a)
¾ IN. NIPPLES AND ELLS											
Electrogalvanized M1	1.60	TrR	0	0-TrY	0-TrY					(a)	(a)
Electrogalvanized C1	2.19	2R	10R	10R	10R					(a)	(a)
Electro-galvanized M1	2.65	20R	20R	30R	20R					(a)	(a)
Electrogalvanized C1	1.25	15R	10R	15R	10R					(a)	(a)
Hot-dip galvanized M1	1.82	80Y	90Y	90Y	90Y	30R-60Y	35R-60Y	50R-50Y	35R-60Y	(a)	(a)
Hot-dip galvanized C1	1.44	60Y	60Y	60Y	60Y					(a)	(a)
Hot-dip aluminum M1	3.20	80Y	95Y	95Y	95Y	0-R-TH	0-R-TH	0-R-TH	0-R-TH	(a)	(a)
Hot-dip aluminum C1	2.80	25Y	75Y	25Y	75Y					(a)	(a)
Electric Sherardized M1	0.75	5R-95Y	5R-95Y	5R-95Y	5R-95Y					(a)	(a)
Amaloy M1 and C1	...	1R-99R*	10R-90R*	10R-90R*	10R-90R*	5R-90R*	10R-80R*	10R-80R*	10R-80R*	(a)	(a)
TOWER CLAMPS											
Hot-dip galvanized M1	2.00	10Y	15Y	10Y	15Y	5R-80Y	10R-20Y	15R-20Y	20R-10Y	(a)	(a)
Hot-dip galvanized C1	2.38									(a)	(a)
Hot-dip aluminum M1	3.16	0-D	0	0	0	0-D	0-D	0	0	(a)	(a)
Hot-dip aluminum C1	2.51					0-D	0	0	0	(a)	(a)
1½ BY 1½ BY ⅛-IN. PLAIN STEEL ANGLES											
Electrogalvanized	0.47									0-30R	10-70R
Electrogalvanized	1.09										
Hot-dip galvanized	3.16	0	0	0	0	5Y	5Y-TrR	2R-5Y	2R-10Y	0	0
Hot-dip aluminum	0.89	0	0	0	0	0-D	0	0	0-D	0	2Y
Cadmium-plated	0.12									50R	70R
Electric Sherardized	0.14									100Y	100Y
Gas Sherardized	...									100R-X	100R-X
Lead coated	...	5R-90R*	10R-90R*	10R-90R*	10R-90R*	5R-80R*	10R-90R*	10R-60R*	10R-90R*	100R*	98R*

TABLE I.—REPORT OF INSPECTIONS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC.; EXPOSED TO ATMOSPHERE ON TEST RACKS AT THREE LOCATIONS.—*Continued.*

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; R*, rust spots widely distributed; Y, yellow or orange yellow rust; D, black or dark surface films (soot or dirt); Tr, trace; X, 100 per cent rust listed in previous reports; TH, threads; a, samples mixed; b, average of nipple and ell. Blank spaces indicate samples removed in 1942.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.				Exposure at Sandy Hook, N. J.				Exposure at Key West, Fla.	
		4-17-46 886 Weeks	10-17-46 912 Weeks	4-23-47 938 Weeks	10-16-47 963 Weeks	4-16-46 894 Weeks	10-16-46 920 Weeks	4-22-47 947 Weeks	10-15-47 972 Weeks	4-10-47 951 Weeks	12-29-47 988 Weeks
1½ BY 1½ BY ⅛-IN. COPPER-BEARING ANGLES											
Electrogalvanized	0.76									15R	35R
Electrogalvanized	0.47									80R	90R
Hot-dip galvanized	3.20	Tr-Y	Tr-Y	Tr-Y	Tr-Y	10Y	5Y	5Y	5Y	0	0
Hot-dip aluminum	1.11	0-D	0	0	0	0-D	0	0	0	2R*	4Y
Cadmium-plated	0.65									55R	75R
Electric Sheradized	...									100Y	100Y
Lead coated	...	5R-80R*	5R-95R*	5R-95R*	5R-95R*	5R-50R*	5R-80R*	5R-70R*	5R-80R*	100R*	80R*
NO. 14 GAGE BY 2½-IN. PLAIN STEEL FLATS											
Electrogalvanized	0.45									0-50R	0-90R
Electrogalvanized	0.70										
Hot-dip galvanized	3.16					3R-15Y	5R 15Y	10R-10Y	5R-20Y	0	0
Hot-dip aluminum	0.89						0	0	0	Tr-R	2Y
Cadmium-plated	...									70R	90R
Electric Sherardized	0.58									100Y	100Y
Gas Sherardized	...									100R	100R
Lead coated	...					2R-60R*	5R-60R*	10R-60R*	5R-50R*	100R*	99R*
NO. 14 GAGE BY 2½-IN. COPPER-BEARING STEEL FLATS											
Electrogalvanized	0.29									20-50R	80-100R
Electrogalvanized	0.62										
Hot-dip galvanized	3.20	Tr-Y	Tr-Y	Tr-Y	5Y					Tr-Y	1Y
Hot-dip aluminum	0.47	0	0	0	0					10R*	25Y
Cadmium-plated	...									99R	95R
Electric Sherardized	...									25R-75Y	60R-40Y
Gas Sherardized	...									100R	100R
Lead coated	...	2R-40R*	5R-60R*	5R-60R*	5R-75R*					100R*	95R*
⅝ BY 2½-IN. BOLTS											
Sherardized	0.87									100Y	100Y
Sherardized	0.70									100R-X	100R-X
Hot-dip galvanized	1.40									0	0
Cadmium-plated	...									80R	90R
Electrogalvanized	0.20									100R-X	100R-X

KNOCKOUT BOXES											
Electrogalvanized	0.21									Tr-Y	Tr-Y
Electrogalvanized	0.16									30R	40R
Hot-dip galvanized	1.56	10Y	15Y	10Y	15Y	1R-90Y	3R-90Y	5R-80Y	5R-90Y	2R	0
Cadmium-plated	0.12									100R-X	100R-X
Cadmium-plated	0.19									100R	100R
Electric Sherardized	0.28	100Y	100Y	100Y	100Y					100R-X	100R-X
Gas Sherardized	0.19									100R-X	100R-X
Amaloy	...	60R*	10R-90R*	10R-90R*	10R-90R*	5R-95R*	10R-90R*	10R-85R*	10R-90R*	100R*	100R*
Hot-dip aluminum	1.84	0	0	0	0	0	0	0	0	20R*	15R*
STEEL HINGES											
Cadmium-plated	0.32									100R	95R
Electric Sherardized	0.40									100R-X	100R-X
Gas Sherardized	0.68									20R-80Y	5R-95Y
Electrogalvanized	0.19									90R	100R*
BULLDOG CLAMPS											
Hot-dip galvanized	1.88					30R	40R	50R	70R	0	0
Hot-dip aluminum	1.35	Tr-R	Tr-R	Tr-R	Tr-R	0	0	0	0	20R*	15R*
¼ BY 3-IN. PLAIN STEEL FLATS											
Electrogalvanized	0.76									Tr-Y	Tr-Y
Electrogalvanized	0.45									15R	25R
Hot-dip galvanized	2.49	Tr-Y	Tr-Y	Tr-Y	Tr-Y	10Y	5Y	5Y	5Y	0	0
Hot-dip aluminum	0.90	0	0	0	0	0	0	0	0	Tr-Y	2Y
Cadmium-plated	0.23									60R	80R
Lead coated	...	1R-90R*	10R-90R*	10R-90R*	10R-90R*	5R-50R*	5R-80R*	5R-70R*	5R-80R*	100R*	98R*
¼ BY 3-IN. COPPER-BEARING STEEL FLATS											
Electrogalvanized	0.84									5Y	15Y
Electrogalvanized	0.66	25R	40R	50R	30R						
Hot-dip galvanized	2.67	Tr-Y	Tr-Y	Tr-Y	Tr-Y	TrR-5Y	TrR-10Y	2R-5Y	2R-10Y	0	0
Hot-dip aluminum	0.66	0	0	0	0	0	0	0	0	0	2Y
Cadmium-plated	0.27									80R	95R
Lead coated	...	1R-60R*	5R-95R*	5R-95R*	5R-95R*	5R-35R*	10R-80R*	10R-70R*	10R-80R*	100R*	98R*
NO. 209 WINDOW FRAME SECTION											
Electrogalvanized	0.67									15R	30R
Electrogalvanized	0.24									15R	60R
Hot-dip galvanized	3.10									0	0
Hot-dip aluminum	0.61									5R*	10R*
Cadmium-plated	0.24									95R	95R
Electric Sherardized	0.52									100Y	100Y+R
Amaloy	...									100R*	95R*
NO. 792 WINDOW FRAME SECTION											
Electrogalvanized	0.54									5R	30R
Electrogalvanized	0.35									50R	95R
Hot-dip galvanized	2.60	0	0	0	0	Tr-R, 20Y	2R-10Y	3R-10Y	2R-20Y	0	0
Hot-dip aluminum	1.39	0	0	0	0	0	0	0	0	TrR-Y	2Y
Cadmium-plated	0.22									40R	75R
Cadmium-plated	0.17									90R	98R
Electric Sherardized	0.57									100Y	98Y
Gas Sherardized	0.53									100R	100R
Parkerized	...									100R-X	100R-X

Less rusting of the basis metal was observed than at the northern locations. All remaining sherardized specimens (gas and electric) are completely covered with red or yellow rust, with difficulty being experienced in differentiating between them. This rust is very smooth and it is believed that considerable zinc of the coating still remains. All remaining cadmium-plated specimens are 50 to 100 per cent rusted. Approximately one quarter of them were reported as completely rusted.

The hardware samples were moved to a new test location during the early part of 1948 through the cooperation of Messrs. Hocker and Taylerson. Samples of doubtful identity or which had been reported previously as completely rusted were discarded in the arrangement of samples on the new racks.

This report has been submitted to letter ballot of Subcommittee XVI, consisting of 25 members; 17 members returned their ballots, of whom 17 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the subcommittee,

LEO J. WALDRON,
Chairman.

REPORT OF COMMITTEE A-6*

ON

MAGNETIC PROPERTIES

Committee A-6 on Magnetic Properties has held two meetings since the previous report, one at Atlantic City, N. J., on June 19, 1947, and the other at Washington, D. C. on March 5, 1948.

The committee is very sorry to report the death of one of its most valued members, K. L. Scott, representative of the Western Electric Co. He was also a representative of the National Electrical Manufacturers Association on Committee A-6. Mr. J. E. Dillinger has been appointed to replace Mr. Scott as the Western Electric representative. G. H. Cole of the American Rolling Mill Co., H. W. Lamson of the General Radio Co., and E. J. Greene of the Bureau of Aeronautics, have all joined the committee during the past year. R. E. Watson of the Leeds & Northrup Co., replaces P. H. Dike.

R. L. Sanford is secretary of Subcommittee No. 1 of the Sectional Committee C42 on Definitions of Electrical Terms, under the sponsorship of the American Standards Association, which deals with fundamental electrical terms including magnetic definitions.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, R. L. Sanford.
Vice-Chairman, B. M. Smith.
Secretary, J. P. Barton.

The committee has recently approved new by-laws governing its procedures. It has also approved new dimensional tolerance values for test strips of electrical sheet.

An extensive revision of the Standard Methods of Testing Magnetic Materials (A 34 – 47) is being planned under a new temporary subcommittee, to be headed by R. C. Taylor of the Western Union Telegraph Co. It is proposed to use A 34 as the designation for the general magnetic test methods, nomenclature, and materials specifications. The A.S.T.M. serial numbers A 340 to A 349 are being reserved and will be assigned to separate sections as issued. For example, the designation A 340 will probably be used for the standard definitions of terms with symbols, A 341 for magnetic test methods for normal induction and hysteresis; etc. The various sections will be issued separately and may also be published collectively.

A Symposium on Magnetic Testing and Properties, sponsored by Committee A-6, has been arranged for the Annual Meeting of the Society in June at Detroit. It will consist of the following nine papers divided into two sessions. It is planned to make these papers available later in the year as a separate publication.

"Testing Magnetic Materials," by B. M. Smith.

"Permanent Magnet Testing Methods and Their Validity in Determining Product Performance," by C. A. Maynard and J. E. Mitch.

"Core Loss Test for Narrow Silicon Steel Strip," by J. A. Ashworth.

"D.-C. Permeability Testing of Epstein Samples with Double-Lapped Joints," by D. C. Dieterly.

"Variation of Core Loss and Permeability of Electrical Grade Silicon Sheet Steel," by A. C Beiler and P. L. Schmidt.

"The Evaluation of Hysteresis Core Loss by Power Equations," by Horatio W. Lamson.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

"Magnetic Analysis Inspection in the Steel Industry," by Theodore Zuschlag.

"Magnetic Stress Analysis," by P. E. Cavanagh and T. Wlodek.

"The Testing of Magnetic Recording Media," by D. E. Weigand.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Permeability of Magnetic Materials (A 34 – 47 T) be approved for reference to letter ballot of the Society for adoption as standard without revision, since no adverse comments have been received with reference to these test methods.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following two standards, as indicated, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Methods of Testing Magnetic Materials (**A 34 - 47**).—Change the symbol for mutual inductance from "M" to "L_m" wherever it occurs in these methods. This change is in line with the recommended change in Standard Definitions of Terms with Symbols, Relating to Magnetic Testing (A 127 – 46) referred to later in this report.

Section 5.—Change the specified dimensions from "± 0.05 cm. (1/64 in.)" to read "± 0.08 cm. (1/32 in.)."

Section 8.—Add a new paragraph (*e*) to read as follows:

(*e*) Test samples from flat-rolled material shall be composed of strips wherein the cross-grain strips are interleaved with the with-grain strips, that is, cross- and with-grain strips are to be alternated. When tested in the Fahy Simplex permeameter, the strip in contact with the magnetizing yoke hall be a cross-grain strip. The number of strips in the test sample shall be a multiple of four in accordance with the following table:

Thickness, in.	Gage Number	Number of Strips
0.0100 to 0.0250...	32 to 24, incl.	12
0.0280 to 0.435....	23 to 19, incl.	8
0.0500 and over....	18 and thicker	4

In order to assist those who wish to construct apparatus for making the 25-cm. Epstein, double-lap joint tests, the following specific information is being supplied concerning the various test equipment components:

Figure 3.—Add the following information for Item 6 to the table under this figure:

Item	Low-Current Mutual Inductor
6. Approximate electrical characteristics:	
Primary inductance.......	5.01 mh.
Primary resistance........	0.248 ohms
Secondary inductance.....	7.35 henrys
Secondary resistance......	860 ohms
Mutual inductance........	181.2 mh.

Item	High-Current Mutual Inductor
6. Approximate electrical characteristics:	
Primary inductance.......	0.14 mh.
Primary resistance........	0.023 ohms
Secondary inductance.....	1.65 henrys
Secondary resistance......	296 ohms
Mutual inductance........	12.9 mh.

Section 20 (*b*).—Add the following as a new Item (*3*):

(*3*) The approximate electrical characteristics to be expected when using the wire sizes specified in the following note are as follows:

Magnetizing winding:	
Resistance...............	0.55 ohms
Inductance...............	1.03 mh.
Potential Winding:	
Resistance...............	1.92 ohms
Inductance...............	10.26 mh.
Mutual Inductance..........	664 microhenrys

In the note following Item (*2*) of Paragraph (*b*), delete "single silk covered" wherever it occurs.

Section 20 (*h*).—Change to read as follows:

(*h*) *Compensating inductor.*—An air-core mutual inductor is required only when the test specimen weighs less than 1 kg. It is used to compensate for the air flux. It may be used permanently, however, for all test specimen weights, in which case the intrinsic induction, B_i, is used (see Note, Section 22(*d*)). Typical specifications for the compensating inductor are given in the table in Fig. 5. The mutual inductance of the compensating inductor is to be the same value as that of the test frame, to within one secondary turn of the compensating inductor. The inductor may be located in the center of the space enclosed by the four coils of the test frame, with the axis of the inductor perpendicular to the plane of the test frame coils.

Figure 4.—Substitute the accompanying Fig. 1 for the present Fig. 4.

New Figure.—Add the accompanying Fig. 2 as a new Fig. 5 and renumber the subsequent figures accordingly.

Section 61 (*a*).—Revise this section to read as follows in order that the maximum height of stack may be kept at approximately 1.5 in. for each range of sheet thickness:

61. (*a*) The test specimen shall consist of the number of strips prescribed in Table II (the accompanying Table I). Each strip shall have a minimum length of 25 cm. ($9\frac{27}{32}$ in.) and a minimum width of 3 cm. ($1\frac{3}{16}$ in.) see Section 5 (*a*)). The strips shall be selected as representative of the surface condition; that is, an equal number of strips should be chosen parallel and perpendicular to the rolling direction, unless otherwise specified.

Section 71.—Change the radius of the ram head of the Erichsen testing machine from "0.95" to read "1.0" cm.

Standard Definitions of Terms, with Symbols, Relating to Magnetic Testing (**A 127 – 46**):

New Symbols.—Change the symbol for mutual inductance from "M" to "L_m" in both Parts I and II and add the following new symbols at the appropriate places to the list in Part I and insert B_{rs} and H_{cs} in the appropriate definitions in Part II:

Symbol	Term
B_m	Maximum induction in a hysteresis loop
H_m	Maximum magnetizing force in a hysteresis loop
B_{rs}	Retentivity
H_{cs}	Coercivity

Change the definition for aging-magnetic to read as follows:

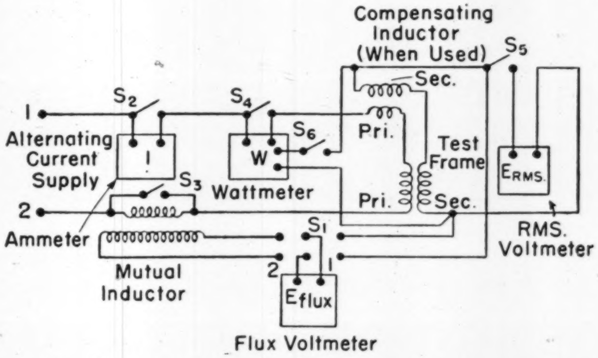


FIG. 1.—Circuit for Core Loss and Permeability Tests.

TABLE I.—NUMBER OF STRIPS IN LAMINATION FACTOR TEST SPECIMEN.

Thickness, in.	Electrical Sheet Gage Number	Number of Strips per Test Specimen
0.0625, 0.0560	16, 17	24
0.0500, 0.0453	18, 19	30
0.0375, 0.0340	20, 21	40
0.0310, 0.0280	22, 23	50
0.0250, 0.0220	24, 25	60
0.0185, 0.0170, 0.0155	26, 27, 28	80
0.0140, 0.0125, 0.0100	29, 30, 31	100

Aging-Magnetic.—The normal change in the magnetic material resulting from gradual metallurgical change in the material. The term also applies when the metallurgical changes are accelerated by increase or decrease in temperature.

NOTE.—When used with reference to core loss, this term, unless otherwise modified, implies an increase in loss.

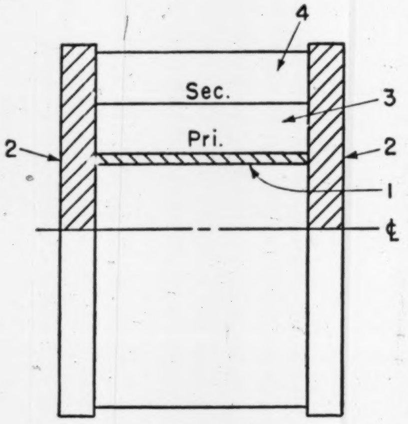


FIG. 2.—Cross-sectional Diagram of Compensating Inductor.

Item	Compensating Inductor
1. Tubular or solid winding form[a]	2 in. in diameter by 1 in. in length.
2. Two end disks[a]	each 4½ in. in diameter by ¼ to ½ in. in thickness.
3. Bottom primary winding	44 turns of 0.072 in. diameter copper wire.
4. Top secondary winding	315 turns of 0.0359 in. diameter copper wire.
5. Approximate electric characteristics:	
Primary inductance	112 microhenrys
Primary resistance	0.06 ohms (approximate)
Secondary inductance	9.8 millihenrys
Secondary resistance	2.22 ohms (approximate)
Mutual inductance	664 microhenrys

[a] The tubular winding form and the end disks shall be made of nonmagnetic, nonconducting material. Other parts of the winding form shall be nonmagnetic.

Reword the definition for demagnetizing coefficient as follows:

Demagnetizing Coefficient, D_B.—Is defined by the equation:

$$D_B = \frac{H_1 - H}{B_i}$$

where: H_1 = Applied magnetizing force as measured in vacuum,
H = magnetizing force actually existing in the magnetic material, and
B_i = intrinsic induction.

Add the following new definition of "magnetic instability:"

Magnetic-Instability.—The change in the condition of magnetization of a magnetic material which may occur gradually, or rapidly, as the effect of temperature, mechanical shock, or external magnetic fields.

NOTE.—These may be reversible changes, or may include the irreversible effects of metallurgical changes.

Permeability Space.—Omit the words "The factor that expresses" from this definition.

Remanence.—Mark this definition with an asterisk.

Symmetrically Cyclically Magnetized Condition.—In the next to the last line, change "so that" to "and."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (P. H. Dike, chairman) held one meeting during the past year. This subcommittee prepared a number of revisions in connection with changes noted earlier in this report under revisions of standards.

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—At a recent meeting of this subcommittee, the chairman presented a very good summary of d–c. magnetic testing methods, which will be submitted in a more concise form for use in connection with the revision of Methods A 34.

Work is still continuing on the investigation of the 25-cm. Epstein specimen for d–c. permeability testing, but no specification has yet been formulated.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—The work of this group for the year has consisted chiefly in minor additions and changes in existing standards, as noted earlier in this report.

Subcommittee V on Coordinating Magnetic Testing Equipment and Methods (J. A. Ashworth, chairman).—A number of 25-cm. Epstein specimens of widely varying characteristics are being collected and tested by a number of suppliers' and users' laboratories throughout the country. It is hoped that by the time the revision of Methods A 34 is completed, more precisely accurate figures for various test methods may be made available.

This report has been submitted to letter ballot of the committee, which consists of 33 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

REPORT OF COMMITTEE A-7*

ON

MALLEABLE IRON CASTINGS

Committee A-7 on Malleable-Iron Castings held two meetings during the year: the first at Atlantic City, N. J., on June 18, 1947, and the second in Washington, D. C., on March 2, 1948.

During the year, 15 new members were added to the committee and there were 2 removals. At the present time the committee consists of 60 members, of whom 56 are voting members; 30 are classified as producers, 16 as consumers and 14 as general interest members.

The present officers of the committee have been re-elected for the ensuing term of two years.

REVISION OF TENTATIVE

The committee recommends the following revisions in the Tentative Specifications for Pearlitic Malleable Iron Castings (A 220 – 47 T):

Sections 4 and 5.—Change to read as follows:

4. (*a*) At the option of the manufacturer and by agreement with the purchaser, machined test bars taken from any convenient coupon and machined according to Fig. 1 may be used. Unless otherwise agreed upon, cast test bars conforming to Fig. 2 or Fig. 3 shall be used.

(*b*) The manufacturer shall cast three or more test specimens from each heat in batch melting and at least every four hours of multiplex melting without chills, using heavy risers of sufficient height to secure sound bars. All specimens shall be suitably identified with the heat number in batch melting and with the pour period in multiplex melting.

(*c*) All test specimens shall be heat treated in regular production equipment as nearly as possible with castings which were made during the same operating heat or period.

5. (*a*) The manufacturer shall test at least one heat treated tension test specimen from each heat of batch melting or from each pour period of multiplex melting and shall record the results, except those of flawed bars, in permanent records. Such records covering the operating periods during which the purchaser's castings were produced should be available for examination by the purchaser's representative.

(*b*) The purchaser's representative may select and test tension specimens representing the approximate operating period during which the purchaser's castings were produced. The test results of flawed bars shall be discarded and duplicate bars shall be tested.

(*c*) Instead of conducting tests as provided in Paragraph (*b*), the representative of the purchaser may elect to examine the manufacturer's test records for the approximate period during which the purchaser's castings were produced. He may further request certified copies of any representative tests which he may select.

(*d*) At the option of the purchaser or his representative, a casting of each design ordered may be tested to destruction, or otherwise broken up, to determine the presence of any manufacturing condition which may be detrimental to the serviceability of the castings.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Specifications for Malleable Iron Castings (A 47 – 47) and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Section 5.—Revise Paragraph (*b*) and add a new Paragraph (*c*) to read as follows:

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

(*b*) The manufacturer shall cast three or more test specimens from each heat in batch melting and at least every four hours of multiplex melting without chills, using heavy risers of sufficient height to secure sound bars. All specimens shall be suitably identified with the heat number in batch melting and with the pour period in multiplex melting.

(*c*) All test specimens shall be annealed in regular production annealing ovens as nearly as possible with castings which were made during the same operating heat or period.

Section 6.—Delete Paragraph (*d*) and revise Paragraphs (*a*), (*b*), and (*c*) to read as indicated above for Paragraphs (*a*), (*b*), and (*c*) of Section 5 of Tentative Specifications A 220, except that the test specimen shall be "annealed" instead of "heat treated."

Section 7 (*a*).—Delete the phrase "but not exceeding $\frac{5}{8}$ by $\frac{3}{4}$ in. in cross-section" at the end of the first sentence. In the last sentence, pluralize "casting" and change "points" to "locations."

Section 7 (*b*).—Change to read as follows:

(*b*) At the option of the purchaser or his representative, a casting of each design ordered may be tested to destruction, or otherwise broken up, to determine the presence of any manufacturing condition which may be detrimental to the serviceability of the casting. The fracture through any heavy sections of this casting may be examined for the presence of primary graphite referred to in Section 3.

Section 8—Change to read as follows:

8. Any castings rejected for improper annealing may be reannealed once. The reannealed castings shall be inspected and if the remaining test lugs, or castings broken as specimens, show the castings to be properly annealed, they shall be accepted; if not, they shall be finally rejected.

Sections 10 and 11.—Substitute the following for these two sections, renumbering the subsequent sections accordingly:

10. The castings shall be free from injurious defects. They shall conform substantially to the patterns or drawings furnished by the purchaser, and to gages which may be specified in individual cases.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

During the current year, Committee A-7 has given consideration to two specific suggestions which were made at the time of the committee meeting in June, 1947.

One of these suggestions was in the form of a recommendation that additional study be made of the Tentative Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts (A 277 – 44 T), the wording of one section of which has been the subject of discussion over a period of several years. A special subcommittee (George E. Bean, chairman), which was then appointed to review the specifications, reported at the Washington Meeting in March, 1948, recommending approval of the specification as now worded and its continuation as tentative. By a subsequent vote of the main committee this recommendation was approved.

The other idea was that recognition of continuous pouring operations and provisions for inspection methods suitable for this form of production procedure should be considered for incorporation in the Standard Specifications for Malleable Iron Castings (A 47 – 47) and in the Tentative Specifications for Pearlitic Malleable Iron Castings (A 220 – 47 T). It so happened that a representative group of Committee A-7 members had been working on this same subject in conjunction with members of the Association of American Railroads, and the results of their deliberations were found to fit in very happily with Committee A-7 requirements. At the Washington meeting the committee accepted for insertion in Specifications A 47 and A 220 the recommendations which were offered on the basis of this joint investigation work. The incorporation of the suggested changes in Specifications A 47 as proposed by Subcommittee III (J. H. Lansing, chairman) and in Specifications A 220 as offered by Subcommittee IV (C. F. Lauenstein, chairman) resulted in the modification of these two specifications referred to earlier in this report.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

During the year no action has been taken by Subcommittees I, II, and V.

This report has been submitted to letter ballot of the committee, which consists of 56 voting members; 31 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

JAMES H. LANSING,
Secretary.

REPORT OF COMMITTEE A-9*
ON
FERRO-ALLOYS

Committee A-9 on Ferro-Alloys was relatively inactive during the war and had held no meetings since March, 1942. A meeting to reactivate the work of the committee was held in Washington, D. C., on March 3, 1948, during the spring group committee meetings of the Society.

Since the last report of Committee A-9, jurisdiction over the Tentative Method for Sampling Molybdenum Salts and Compounds for Metallurgical Use (E 66 – 34 T) was transferred by mutual consent and the approval of the Board of Directors from Committee E-3 on Chemical Analysis of Metals back to Committee A-9 which had originally written this method under the designation A 156.

At the meeting in Washington in March, 1948, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. C. Bowden, Jr.
Vice-Chairman, C. M. Loeb, Jr.
Secretary, G. F. Comstock.

Mr. Bowden was also elected to fill the unexpired term of J. H. Critchett who had retired during 1947.

Reaffirmation of Standards

The committee recommends that the following standards be reaffirmed and continued in their present status without change:

Standard Specifications for:
Spiegeleisen (A 98 – 41),
Ferromanganese (A 99 – 39),
Ferrosilicon (A 100 – 39),
Ferrochromium (A 101 – 42),
Ferrovanadium (A 102 – 39),
Ferromolybdenum (A 132 – 39), and
Ferrotungsten (A 144 – 39).

Activities of Subcommittees

Subcommittee I on Specifications (E. A. Lucas, Chairman) has been instructed by the committee to prepare specifications covering ferrotitanium and ferroboron and to revise the Standard Specifications for Molybdenum Salts and Compounds (A 146–39) including therein the material now in Method E 66.

Committee A-9 hopes to cooperate with Committee B-9 on Metal Powders and Metal Powder Products in writing specifications for tungsten, molybdenum, etc. Any action on this must await an examination of the scope of the two committees involved.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. C. Bowden, Jr.
Chairman.

A. P. Spooner,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

REPORT OF COMMITTEE A-10*

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held meetings at Atlantic City, N. J., on June 17, 1947, during the Annual Meeting of the Society, and on March 2, 1948, at Washington, D. C.

Some changes in representation on the committee occurred during the past year. The present committee consists of 93 members, of whom 41 are classified as producers, 37 as consumers, and 15 as general interest members. H. L. Maxwell, vice-chairman of Committee A-10, found it necessary to resign from committee activities.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Jerome Strauss.
Vice-Chairman, L. L. Wyman.
Secretary, H. D. Newell.

There are a number of tentatives under the jurisdiction of this committee which, by the rules of the Society, should be reviewed to determine if they should be adopted as standard. In each case it develops that there may be revisions to be made in these specifications during the next year so that the committee at this time is not recommending adoption of any of these tentatives as standard. Details of proposed work on the specifications will be noted under Activities of Subcommittees.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (F. P. Peters, chairman) held a formal meeting on March 2, 1948, at which time the status of the publication projects which the committee has under way were examined and plans were made for bringing them to the earliest possible fruition. The data on the cast corrosion-resisting and heat-resisting alloys have been completed. As soon as a revision of the data on the wrought alloys (previously published by the committee in 1942) is completed in collaboration with a special committee of the American Iron and Steel Institute, the two sets of data will be combined and published by A.S.T.M. as Tables of Data on Chemical Compositions, Physical and Mechanical Properties of Wrought and Cast Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Alloys.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman) has been actively engaged on the following projects:

1. Continued development of plans for atmospheric corrosion tests which will be started when new test locations being developed by the Advisory Committee on Corrosion are available.

2. Inspection of a number of stainless steel architectural structures, especially at Atlantic City, N. J., as described in the Appendix.

3. Continuation as tentative, pending contemplated revision, of the Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion Resisting Steels (A 262 – 44 T)[1] and the Tentative

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1946 Book of A.S.T.M. Standards, Part I-A.

Method of Total Immersion Corrosion Test of Stainless Steels (A 279 – 44 T).[1]

4. Arrangement for a subgroup to prepare a discussion of effects of methods of preparation of surface of test specimens to be added to Tentative Method A 279.

5. Recommendation that A.S.T.M. stimulate a broad research program on the fundamentals of stress-corrosion cracking of material. An outline of the proposed research and the reason therefor have been brought to the attention of the Advisory Committee on Corrosion and to the Administrative Committee on Research. An initial contact has also been made with the Office of Naval Research seeking to secure support for the program.

6. Initiation of plans for a symposium next year on the behavior of corrosion-resistant steels in respect to comparison between results from present testing procedures and behavior in service combined with the influence of very low-carbon content in the austenitic types.

Subcommittee V on Mechanical Testing (T. F. Olt, chairman) met in Washington on March 1, 1948, and organized a fourth round-robin schedule of tension testing of austenitic grades in which six laboratories will cooperate. The first three round-robin tests were not entirely to the satisfaction of the committee, as there has come into the question the possible necessity of maintenance of an extremely uniform temperature during the testing, which may prove to be even more important than the rate of strain. This is particularly true in the measurement of the elastic properties of these materials.

A request has been forwarded to the Section on Tension Testing, of Committee E-1 on Methods of Testing, to consider an editorial addition to the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46)[1] which would qualify the use of the 10-in. gage length wire specimen. This subcommittee has asked Subcommittee IX to review Standard Specifications for Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (A 176 – 44)[1] and the Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 44)[1] and include the 0.2 per cent offset yield strength, retaining the 0.5 per cent extension under load as an approved alternate method.

Subcommittee VI on Metallography (R. Franks, chairman) has continued its project on methods of identifying the sigma phase that develops in certain austenitic chromium-nickel steels, and has progressed to a point where all of the steels for the test program have been collected. Samples are being prepared for distribution to cooperating laboratories which will make impact tests before and after holding at the high temperatures which bring about the sigma formation. At the last meeting of the subcommittee the complete program for distributing the samples for sigma phase identification was laid out.

Subcommittee VIII on Specifications for Wrought Products (J. K. Findley, chairman) has under consideration a revision of the chemical composition requirements in Specifications for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 – 44 T),[1] endeavoring to correlate these with the latest A.I.S.I. compositions.

Subcommittee IX on Specifications for Flat Products (H. A. Grove, chairman) has been considering alternate tension test specimens for plates in Specifications A 167, A 176, and A 240 and is proposing as an alternate a standard 8-in. gage length flat specimen as shown in Fig. 1 of Methods E 8 – 46. The subcommittee has also had under consideration the question of including type 405 and type 316-Cb alloys in Specifications A 240 subject to the approval of the A.S.M.E. Boiler Code Committee.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman) has under review certain differences between chemical composition of certain grades in the two general casting Specifications A 296[1] and A 297[1] and the corresponding alloy compositions published by the Alloy Casting Institute. It is expected that reconciliation can be accomplished between A.S.T.M. and A.C.I. during the current year and the two specifications adopted as standard in 1949. The subcommittee is also confronted with the problem of correlating the A.S.T.M. designations for alloy grades in the specifications with the A.I.S.I. designations used for wrought alloys, particularly with reference to applications where castings are to be joined to wrought parts. This problem is now under consideration jointly with representatives of the A.I.S.I. Stainless Steel Committee.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman) has been working during the past year with Committee A-1 on Steel in the preparation of Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe.[2] This will be completed shortly after the annual meeting and submitted to the Society through the Administrative Committee on Standards. This specification was prepared to fill a need for welded and seamless stainless pipe for general service and also covers high-temperature piping of the austenitic grades which have formed a part of Specification A 158.

This report has been submitted to letter ballot of the committee, which consists of 93 members; 70 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

---

[2] See Editorial Note, below.

EDITORIAL NOTE

On November 15, 1948, the Administrative Committee on Standards accepted the joint recommendation of Committees A-10 and A-1 that the Specifications for Seamless and Welded Austenitic Stainless Steel Pipe be approved for publication as tentative. The new tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-A, bearing the designation A 312 – 48 T.

APPENDIX

REPORT OF SPECIAL SUBCOMMITTEE ON STAINLESS STEEL ARCHITECTURAL STRUCTURES

In June, 1947, a special group of Subcommittee IV inspected several installations of stainless steel in store fronts on the Boardwalk at Atlantic City, N.J. An earlier inspection of the stainless steel used in store fronts on the Boardwalk at Atlantic City, made in June, 1938, was discussed in the Report of the Special Subcommittee on Inspection of Stainless Steel Architectural Structures.[1]

Fifteen installations were examined, all located directly on the Boardwalk and, therefore, subjected to the action of sea spray and mist. Some of these store fronts were several years old.

The inspection group included the following:

H. Huester, U. S. Navy
Russell Franks, Electro Metallurgical Corp.
F. L. LaQue, The International Nickel Co., Inc.
V. N. Krivobok, The International Nickel Co., Inc.
M. H. Brown, E. I. du Pont de Nemours & Co.
E. S. Taylerson, Carnegie-Illinois Steel Corp.
H. A. Grove, Republic Steel Corp.

Of the fronts inspected, ten were found to be in good condition. The condition of the remaining five reflected lack of care, since it was found that what rust staining was present could be removed readily to restore the original lustre of the stainless steel.

The installations inspected represented only a few of the many stainless steel store fronts on the Boardwalk, but were considered to provide a representative sample for the purposes of this inspection.

Respectfully submitted on behalf of the special subcommittee,

H. A. Grove,
Chairman.

---

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 200 (1939).

REPORT OF COMMITTEE B-1*

ON

WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Wires for Electrical Conductors held two meetings during the year: one in Atlantic City, N. J., on June 17, 1947, and one in New York, N. Y., on March 19, 1948. Meetings of the Advisory Committee and of Subcommittees II, IV, VI and VII were also held during the year.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Foote.
Vice-Chairman, W. R. Hibbard.
Secretary, E. H. Kendall.[1]

Election of members-at-large of the Advisory Committee resulted in the selection of the following:

J. R. Becker
G. H. Harnden
W. F. Markley
R. S. Pratt
B. J. Sirois

The subcommittee organization and their chairmen remain unchanged.

Subsequent to the election of officers and the preparation of this report, the committee notes with deep regret the passing on April 17, of Secretary E. H. Kendall. His cheerful, unselfish and unstinted services on behalf of the committee will be sorely missed by all.

The committee now consists of 67 members, of whom 25 are classified as consumers, 33 as producers, and 9 as general interest members.

Subsequent to the 1947 Annual Meeting, there was presented to the Society through the Administrative Committee on Standards the proposed Tentative Classification of Copper which was prepared by a joint committee representing Committee B-1, Committee B-2 on Non-Ferrous Metals and Alloys, and Committee B-5 on Copper and Copper Alloys. The new tentative classification was accepted by the Standards Committee on March 5, 1948, and has been issued under the designation B 224 - 48 T.

I. NEW TENTATIVES

Following the expansion of scope of Committee B-1, authorized two years ago, to include wires for electrical conductors of materials other than copper and its alloys, there has arisen a demand both from consumer and from producer interests for specifications for electrical conductors of aluminum and of a combination of steel with aluminum and with copper, in various forms. Conductors of such materials and constructions have been manufactured for a number of years, but specifications therefor on an adequate and national basis have not been available. The same is true of all-copper conductors stranded in annular form.

The committee, therefore, recommends for publication as tentative the following eight new specifications as appended hereto:[2]

Specifications for:

Rolled Aluminum Rods (EC Grade) for Electrical Purposes,
Cored, Annular, Concentric-Lay-Stranded Copper Conductors,
Hard-Drawn Copper-Covered Steel Wire,
Concentric-Lay-Stranded Copper-Covered Steel Conductors,
Concentric-Lay-Stranded Copper and Copper-Covered Steel Composite Conductors,

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] It was reported at the Annual Meeting that A. A. Jones had been elected secretary to fill the vacancy created by the death of E. H. Kendall.

[2] These specifications were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

Hard-Drawn Aluminum Wire for Electrical Purposes,

Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn, and

Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR).

II. Revision of Tentative

Tentative Method of Test for Resistivity of Copper and Copper Alloy Electrical Conductors (**B 193 - 45 T**).[3]—The committee recommends that this tentative method be revised as appended hereto.[4]

III. Revision of Standards and Reversion to Tentative

The committee recommends the following revisions in two standards and reversion of these revised specifications to tentative:

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard or Soft (**B 8 - 46**):[3]

Section 1.—In the first line of Paragraph (*a*), insert the word "bare" after the word "cover."

In Paragraph (*b*), for class A conductors, in the first line, insert the words "conductors to be covered with" after the word "for." In the third line, substitute the word "materials" for the word "conductors."

For class B conductors, in the first line, insert the words "to be" after the word "conductors."

Section 6.—Add to the section heading the words: "of Conductors Stranded of Soft Wires." Substitute the following for Paragraphs (*a*), (*c*), (*d*), (*e*), and add a new Paragraph (*f*), relettering the present Paragraph (*b*) as (*g*).

6. (*a*) Tests for the electrical properties of wires composing conductors made from soft or annealed copper wire, bare or coated, shall be made before stranding.

(*b*) Tests for the physical properties of soft or annealed copper wire, bare or coated, may be made upon the wires before stranding or upon wires removed from the completed stranded conductor, but need not be made upon both. Care shall be taken to avoid mechanical injury to wire removed from the conductor for the purpose of testing.

(c) The physical properties of wire when tested before stranding shall conform to the applicable requirements of Section 2(*b*).

(*d*) The physical properties of wires removed from the completed stranded conductor shall be permitted to vary from the applicable requirements of Section 2(*b*) by the following amounts (Note 5):

(*1*) *Average of Results Obtained on All Wires Tested.*—The maximum tensile strength permitted shall be 5 per cent greater than the maximum prescribed for the wires before stranding and the minimum elongation required shall be reduced in numerical value 5 (for example, from 30 per cent to 25 per cent) from the numerical requirements for the wire before stranding.

(*2*) *Results Obtained on Individual Wires.*—The tensile strength of individual wires shall not exceed the maximum prescribed before stranding by more than 15 per cent, and the elongation of individual wires shall be reduced in numerical value 15 from the minimum requirements before stranding (that is, 10 in addition to the 5 allowed in Item (*1*)), but in no case shall the elongation of any individual wire be less than 5 per cent.

(*e*) In the event that the requirements prescribed in Item (*2*) of Paragraph (*d*) are met but those prescribed in Item (*1*) are not met, a retest shall be permitted wherein all wires of the conductor shall be tested for the purpose of final determination of conformance to Paragraph (*d*).

(*f*) By agreement between the purchaser or his representative, and the manufacturer, the tensile strength of the completed stranded conductor as a unit may be determined in lieu of but not in addition to physical tests on individual wires before or after stranding. The breaking strength of conductors so tested shall be not greater than 5 per cent higher than the total of the specified maximum breaking strengths of the component wires as set forth in Section 2(*b*). The maximum breaking strength of the component wires shall be calculated using nominal specified diameters and maximum specified tensile strengths. The free length between grips of test specimens shall not be less than 10 in. and should be greater than 10 in. for the larger sizes of conductors. Care shall be taken to insure that the wires in the conductor are evenly gripped during the test (Note 4).

---

[3] 1946 Book of A.S.T.M. Standards, Part I-B

[4] This revised method was accepted by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

New Section 7.—Redesignate present Section 6 (*c*), (*d*), and (*e*) as new Section 7 (*a*), (*b*), and (*c*) under the heading "Physical and Electrical Tests of Conductors Stranded of Hard-Drawn or Medium-Hard-Drawn Wires," and renumber the subsequent sections accordingly.

Standard Specifications for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (**B 48 – 45**):[3]

Section 5(*a*). Change to read as follows:

5. (*a*) Type B wire shall successfully withstand bending edgewise through 180 deg. around a mandrel without cracking under normal conditions of edgewise bending during manufacturing operations. The mandrel diameter shall not be less than one-half the nominal width of the material being bent. In cases where the mandrel diameter used is less than $\frac{5}{32}$ in. or the thickness is less than 0.020 in. or the ratio of width to thickness of the wire is greater than 30 to 1 the scope of class B wire is exceeded and the edgewise bending properties of the wire shall be as agreed upon between the purchaser and the manufacturer (Notes 9 and 10).

Delete Paragraphs (*c*) and (*d*).

Explanatory Notes.—Add the following as new notes 9 and 10:

NOTE 9.—At the present time, no standard definitive test for determining the edgewise bend characteristics of soft rectangular copper wire is available. When such a test has been developed, it is contemplated that Section 5(*a*) of these specifications will be revised to include definite test requirements.

NOTE 10.—It is expected that the product furnished by the manufacturer will withstand the specified edgewise bend during fabrication by the purchaser and that its ability to do so will be determined prior to shipment by means agreed upon between the purchaser and the manufacturer.

IV. ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (**B 189 – 45 T**).[3]—The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard, without revision. These specifications have stood for two years without revision, during which time no criticisms or suggestions for improvement of a material nature have been received.

V. TENTATIVES CONTINUED WITHOUT REVISION

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (**B 172 – 45 T**),[3] *Having Concentric-Stranded Members for Electrical Conductors* (**B 173 – 46 T**),[3] and *for Bunch-Stranded Copper Conductors for Electrical Conductors* (**B 174 – 45 T**).[3]—The committee recommends that these specifications be continued as tentative with slight editorial revisions, as there is a possibility that certain revisions will be recommended during the coming year.

VI. EDITORIAL CHANGES

The committee recommends editorial changes in the following five specifications, comprising the elimination of open ranges in tabulations of size of wire; for example, in Standard B 1, the nominal diameters now specified in Section 4 as "0.460 to 0.325, and 0.324 to 0.0403" will be changed to read: "0.460 to 0.325, incl., and under 0.325 to 0.0403, incl." These changes are of a minor nature and do not affect or constitute material revisions in the requirements. They do, however, clarify, remove inconsistencies, and effect significant improvements in the specifications.

Standard Specifications for:
Hard-Drawn Copper Wire (B 1 – 47),
Medium-Hard-Drawn Copper Wire (B 2 – 47),
Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 46),
Soft Rectangular and Square Bare Copper Wire for Electrical Purposes (B 48 – 45), and

Tentative Specifications for:

Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 – 45 T).

The committee also recommends an editorial change in the following two standards. In the cross-sectional view of the 300,000 cir. mils wire, shown in Fig. 1, change the dimension from the center line of the grooves to the center line of the section from "0.125 in." to read "0.127 in."

Standard Specifications for:

Bronze Trolley Wire (B 9 – 46), and
Copper Trolley Wire (B 47 – 46).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (E. H. Kendall, chairman) reviewed from an editorial standpoint all of the new specifications and revisions of present standards and tentatives referred to earlier in this report. One of the most important assignments pending in this subcommittee, which will now be under the chairmanship of A. A. Jones, is the compilation of a history of the committee's activities.

Subcommittee II on Methods of Test and Sampling Procedure (C. S. Gordon, chairman) completed the extensive revision and expansion of Tentative Method B 193 mentioned earlier in this report. This subcommittee is actively engaged in the project of consolidating and coordinating methods of test used throughout the committee's specifications with special reference to a practical sampling procedure following the pattern to be set by Committee E-11 on Quality Control of Materials.

Subcommittee III on Rods for Processing into Conductors (Alfred Bellis, chairman) prepared the new tentative specifications for aluminum rod for subsequent processing into wire, previously referred to in this report.

Subcommittee IV on Conductors of Copper and Copper Alloys (H. H. Stout, Jr., chairman) has more or less taken over the work that the main committee covered a few years ago. It prepared the new Tentative Specifications for Cored, Annular, Concentric-Lay-Stranded Copper Conductors. It also recommended the actions previously noted with respect to Specifications B 8, B 48, B 172, B 173, B 174, and B 189, as well as the editorial corrections in two of these specifications and in Specifications B 1, B 2, B 9, B 33, and B 47 as noted above.

This subcommittee has been working on a method of checking the shape and dimensions of grooved trolley wire and has arrived at a solution to this problem which has been before the committee since 1912. These recommendations were not completed in time for inclusion in this report but will be submitted later in the year to the Administrative Committee on Standards as tentative revisions of Standard Specifications B 9 and B 47.[6] The subcommittee is also preparing a clause covering the weight-density method for area determination and is reviewing all tolerances. The problem of edgewise bending has been referred to the Coordinating Committee on Non-Ferrous Metals and Alloys to secure cooperation of other committees which are faced with the same problem.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) is working on a specification for the steel wire used for reinforcement of aluminum cable.

Subcommittee VI on Composite Conductors of Copper and Steel (C. E. Ambelang, chairman) completed the three new tentatives involving copper-covered steel wire previously noted. The subcommittee plans to study during the coming year the possibility of preparing a specification covering copper and galvanized steel composite conductors.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[6] See Editorial Note, p. 142.

Subcommittee VII on Conductors of Light Metals (P. V. Faragher, chairman) completed the new tentative specifications for aluminum wire and the two new tentative specifications for aluminum cable previously mentioned in this report. The subcommittee will cooperate during the next year with Subcommittee V in the preparation of the specifications for steel core wires.

This report has been submitted to letter ballot of the committee, which consists of 67 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

E. H. KENDALL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Bronze Trolley Wire (B 9 – 46),
Copper Trolley Wire (B 47 – 46), and
Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 46).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the tentative revisions appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

REPORT OF COMMITTEE B-2* ON NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held one meeting on June 18, 1947, in Atlantic City, N. J., during the Annual Meeting of the Society. Since the June meeting, the work of Committee B-2 has been handled by correspondence.

The Advisory Committee held two meetings: on June 18, 1947, immediately preceding the meeting of Committee B-2, and on March 4, 1948, in Washington, D. C., during the Spring Group Committee Meetings. The Advisory Committee at both meetings approved changes in membership of Committee B-2, and accepted reports of the work of the subcommittees. At the March meeting, the decision of Mr. Thum to retire from the chairmanship of the committee was received with regret. The selection by the Nominating Committee of Bruce W. Gonser for chairman, and G. Howard LeFevre for secretary, was approved. These nominations were presented to Committee B-2 at the 1948 Annual Meeting in Detroit and the nominees were elected for the ensuing term of two years.

The subcommittees having work for consideration held meetings during the 1947 Annual Meeting at Atlantic City and during the Spring Group Committee Meetings in Washington. Business of the subcommittees during the year has been handled by task groups and by correspondence.

Joint Committee on Classification of Copper (Sidney Rolle, chairman; Carter Cole, secretary) of Committee B-1 on Wires for Electrical Conductors, Committee B-2 and Committee B-5 on Copper and Copper Alloys, Cast and Wrought, met on June 19, 1947, in Atlantic City and accepted, with amendments, its task group's report and the proposed classification of copper. This classification was accepted by the Administrative Committee on Standards on March 5, 1948, as the Tentative Classification of Copper (B 224 – 48 T).[1]

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Fire-Refined Copper for Wrought Alloys (B 216 – 46 T).

Revision and Reversion to Tentative of Standard Specifications for:

Fire-Refined Copper Other than Lake (B 72 – 33).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, the following revisions in the Standard Specifications for Slab

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

Zinc (Spelter) (B 6 – 46) and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Table I.—Add the following as a new Note 2 to this table:

NOTE 2.—For purposes of acceptance or rejection, an observed value or calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit, in accordance with the rounding-off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).

Section 6.—Revise to read as follows:

6. Samples shall be selected and prepared in accordance with Sections 3, 4, and 5 of the Tentative Method of Sampling Slab Zinc (Spelter) (A.S.T.M. Designation: E 65).

New Section.—Add the following as a new Section 7 renumbering the subsequent sections accordingly:

7. *Methods of Chemical Analysis.*—The chemical compositions enumerated in these specifications shall be determined by the following methods of the American Society for Testing Materials (in cases of dispute, Methods E 40 shall be used, except for the Special High Grade and the High Grade, where Methods E 26 may be used as an alternate):

	A.S.T.M. Designation[a]
Chemical analysis	E 40
Spectrochemical analysis	E 26
Photometric analysis	E 64
Polarographic analysis	E 68

[a] These designations refer to the following methods of testing:
Standard Methods of Chemical Analysis of Slab Zinc (Spelter) (A.S.T.M. Designation: E 40),
Tentative Methods of Spectrochemical Analysis of Zinc for Lead, Iron, and Cadmium (A.S.T.M. Designation: E 26),
Tentative Photometric Methods for Determination of Iron in Slab Zinc (Spelter) (A.S.T.M. Designation: E 64), and
Tentative Method for Polarographic Determination of Lead and Cadmium in Zinc (A.S.T.M. Designation: E 68).

The above recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (William E. Milligan, chairman) completed work on the correlation of Standard Specifications B 72 and Tentative Specifications B 216 as referred to earlier in this report.

The subcommittees noted with regret the retirement of W. B. Price, and in appreciation of his contributions to the work of Subcommittee I and to non-ferrous metallurgy, unanimously adopted the following resolution:

> Subcommittee I, Refined Copper, of Committee B-2 on Non-Ferrous Metals and Alloys notes with regret the retirement of Mr. W. B. Price as Director of Research of the Scovill Manufacturing Co. on January 1, 1948. We congratulate him on having outstandingly contributed to non-ferrous metallurgy in the fulfillment of his duties and we shall miss him greatly.
>
> Mr. Price was an early member of Subcommittee I of Committee B-2 and from the beginning took an active interest in the affairs of the committee and the Society generally. As a representative of one of the large consumers of refined copper his opinions have been highly respected.
>
> Bill, as his more intimate friends invariably call him, is a genial member of the non-ferrous group and an esteemed member of the renowned Committee X. His cheerfulness, his good nature and his keen sense of humor have always made him a welcome member of any gathering.
>
> Subcommittee I of Committee B-2 takes this occasion to congratulate him on his release from active service and to express the hope that with good health he may continue to enjoy thoroughly and for a long time his new found leisure.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (Sidney Rolle, chairman) is preparing specifications for antimony and has revised the Standard Specifications for Sampling Pig Lead (B 29 – 43). The recommended revision is being reviewed by Committee E-3 on Chemical Analysis of Metals. With regard to the necessity for a specification for tin, the subcommittee authorized the preparation of a Report on Tin by Marshall Tuthill, as appended hereto.[3]

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[3] See p. 146.

Subcommittee III on White Metals and Alloys (G. H. Clamer, chairman) made minor changes in the Tentative Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 46 T), and in Tentative Specifications for Soft Solder Metal (B 32 – 46 T). These revisions will be submitted to the Administrative Committee on Standards in the near future.[4]

Investigation has revealed that at this time specifications for type metal and fusible alloys are not desirable.

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman) prepared the revisions of Standard Specifications B 6 covered earlier in this report to bring these specifications in line with the Society's recommended practice and methods of analysis.

Subcommittee V on Precious Metals and Alloys (R. H. Leach, chairman) with approval of the Advisory Committee relinguished jurisdiction over Standard Specifications for Silver Solder (B 73 – 29) to the Joint Committee on Filler Metal, which joint committee is sponsored by the American Welding Society and A.S.T.M. At present there is no work before the subcommittee.

Subcommittee VI on Coated Metals (Griffith Williams, Jr., chairman).—A task group is working on the revision of Standard Specification for Lead-Coated Copper Sheets (B 101 – 40).

Subcommittee VII on Refined-Nickel and High-Nickel Alloys, Cast and Wrought (O. B. J. Fraser, chairman) has in preparation revisions of the Tentative Specifications for Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 41 T) and for Nickel-Copper Alloy Rods and Bars (B 164 – 41 T).

The subcommittee has before it for review in 1948 all of the specifications under its jurisdiction and is also considering writing additional specifications.

Subcommittee VIII on Miscellaneous Metals and Alloys (E. E. Schumacher, chairman) held one meeting during the year for the purpose of discussing proposed specifications for cadmium which might be requested by the Treasury Department but as there was no interest in such specifications by the Treasury Department, no action was taken by the subcommittee. This confirms the action of the subcommittee at its meeting February 27, 1947, in Philadelphia, when at that meeting of producers and consumers it was decided that a specification was not necessary or desirable.

This report has been submitted to letter ballot of the committee, which consists of 140 members; 77 members returned their ballots, of whom 69 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

G. HOWARD LEFEVRE,
Secretary.

---

[4] See Editorial Note.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:
Soft Solder Metal (B 32 – 46 T), and
White Metal Bearing Alloys (B 23 – 46 T).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the revised tentatives appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

APPENDIX

A REVIEW OF THE TIN SITUATION

BY MARSHALL W. TUTHILL[1]

This general review of the tin situation is offered with the view of assisting Committee B-2 in determining whether or not it is desirable for the Society to issue specifications for tin. Two previous reviews of the tin situation have been published as appendices to the reports of Committee B-2. The last of these, by Lawrence Addicks,[2] was in 1923 and was but a slight revision of an earlier report[3] published in 1916.

Specifications:

Throughout the recent war, and continuing into the postwar period, the U. S. Government procured, stored, and distributed tin, mostly designated as Grade A. There are at present three Government specifications for tin. The most important of these is the specification for Grade A tin, issued by the Treasury Procurement Division under the designation U. S. Specification S-14. This specification is the basis for the Government contract for Grade A tin with the Tin Processing Corp.'s smelting plant at Texas City, Tex. The second is Federal Specification QQ-T-371a, last revised in 1944 and covering also only a Grade A tin. Third, last revised in 1924, and not recently used, is the Navy Department Specification 46T1d. Two grades, A and B, are included in this specification. The requirements of all three specifications are set forth in Table I.

Tin outside of the limits for Grade A is now usually sold on its actual analysis and diverted to uses where its lower quality is not detrimental. These discount grades, which originally were based upon the now obsolete OPA Regulation M.P.R. No. 17, vary from time to time as regards tin content, the present specifications being as follows:

Grade B—99.80 per cent or higher, which does not meet specifications for Grade A, providing arsenic content does not exceed 0.05 per cent.

Grade C—99.65 per cent to 99.79 per cent and also 99.80 per cent or higher, which does not meet the specifications for Grade A or Grade B.

Grade D—99.50 per cent to 99.649 per cent.

Grade E—99.00 per cent to 99.499 per cent.

Grade F—98.00 per cent to 98.999 per cent.

Grade G—95.00 per cent to 97.999 per cent.

Allowable Impurities:

The impurities stipulated under U. S. Specification S-14, as set forth in Table I, may be taken as the maximum percentages allowable for tinplate. Tinplate makers have found that excess lead tends to make a mottled finish, excess iron causes a rough surface and impairs fluidity, and excess arsenic and zinc tend to form poisonous salts. In making solder, a higher lead content is acceptable, and in bronze a higher iron content (up to 0.05 per cent) is not objectionable.

---

[1] Tuthill & Co., Inc., New York, N. Y.

[2] Lawrence Addicks, "A Review of the Tin Situation," *Proceedings*, Am. Soc. Testing, Mats. Vol. 23, Part I, p. 184 (1923).

[3] "A Review of the Tin Situation," *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part I, p. 187 (1916).

Brand Names:

For a great many years, tin metal has been marketed by brands, and consumers have bought by brand to suit their varying needs: namely, as a noncorrosive, ductile, nontoxic coating on steel (tinplate); as an anti-friction metal (bearing and babbitt metals); or for use in an alloy, as in solder, bronze, brass and so forth. By far the larger part of the tin used is Grade A, and the following brands of pig tin are classified as acceptable under this designation as can be seen by reference to the average analyses of the various brands given in Table II.

TABLE I.—GOVERNMENT TIN SPECIFICATIONS.
(All percentages are maximum values except in the case of tin which is a minimum)

	Tin	Antimony	Arsenic	Lead	Bismuth	Copper	Iron	Silver	Sulfur	Cobalt plus Nickel	Cadmium	Zinc
U. S. Spec. S-14 (Grade A)	99.80	0.040	0.050	0.050	0.015	0.040	0.015	0.001	0.010	0.010	0.001	...
QQ-T-371a (Grade A)	99.85	0.050	0.040	0.050	0.015	0.040	0.015	...	0.010	...	...	[a]
46Tld (Grade A)	99.75	0.10	0.10	0.10	0.01	0.10	0.01	...	0.01	...	[a]	[a]
46Tld (Grade B)	98.0	1.50	0.10	1.50	0.10	0.10	0.10	...	0.01	...	...	[a]

[a] None as determined by a 20-g. sample.

Banka
Billiton
Chempur
E. S. Coy (Penang)
Hawthorne Refined
Katanga
O. T. Lempriere & Co.
Longhorn 3 Star
M. & T. Electrolytic Refined
Mellanear (Guar. 99 per cent)
Pass No. 1
Pyrmont
Straits Trading
Vulcan American Refined
Vulcan Electrolytic

Sources:

By far the larger part of the world's tin ore comes from that long belt running southward from China through Siam and the Malay Archipelago through the Netherlands East Indies and then on down to Australia. From the alluvial deposits of the Far East, tin ores are recovered by bucket and cutter dredging, pumping, panning and ground sluicing. During the recent world war, the larger part of these areas was in the hands of the Japanese. The dislocation of war is reflected in the tin production figures in Table III.

The lode mines, as in Bolivia, call for deep tunneling and the use of explosives, drills, and equipment common to such operations. The larger part of Bolivia's ore production was smelted in Great Britain and on the Continent until the Longhorn tin smelter at Texas City, was built. During 1940 to date, American imports of Bolivian ore totalled 174,652 long tons of contained tin. During this same period, the Belgian Congo supplied the United States with 26,635 long tons of contained tin in ore and 62,174 tons of tin metal. It was indeed fortunate for America's war-time economy that these sources of supply were kept open, for the Governmental and industrial stockpile of tin at the time of Pearl Harbor would have lasted less than two years, even with the rigid conservation orders and despite the stepped-up tin metal imports of 1940 to 1942, which imports were as follows:

From British Malaya:	209,117 long tons
From Netherlands Indies:	33,762 long tons
From China:	10,359 long tons
From United Kingdom:	8,579 long tons

Japanese War Production:

During the occupation years of 1942 to 1945, the Japanese operated several

Far Eastern smelters and produced a very considerable tonnage of pig tin. That smelted in the Netherlands Indies was cast into the regular Banka molds. In their smelting operations in Malaya, the Japanese used special Japanese markings instead of the standard Straits brands. At least 12,000 tons of this Japanese-made tin, recovered in Japan and elsewhere in the Far East, came to the U.S.A. in 1946 and 1947 and was marketed by the R.F.C. on the basis of U. S. assays, as this war-produced tin was characterized by varying quality and sub-standard casting in large part.

TABLE II.—TYPICAL TIN ANALYSES.

Sources: *Metal Statistics*, 1947; *The American Metal Market;* The Vulcan Detinning Co.; Texas City Smelter.

Brand	Chemical Composition, per cent.											
	Tin	Antimony	Arsenic	Lead	Bismuth	Copper	Iron	Silver	Sulfur	Cobalt	Nickel	Cadmium
American:												
Longhorn 3-Star (Grade A)	99.894	0.024	0.013	0.039	0.004	0.020	0.002	trace	0.002	0.002		nil
Longhorn 2-Star (Grade B)	99.835	0.019	0.005	0.120	0.006	0.010	0.001	trace	0.002	0.002		nil
Longhorn 2-Star (Grade C)	99.725	0.026	0.007	0.215	0.010	0.012	0.001	trace	0.002	0.002		nil
M&T Refined	99.978	0.006	trace	0.055	0.0005	0.0009	0.009	nil	nil	nil	nil	nil
Vulcan Electrolytic	99.9966	0.0005	trace	0.0005	0.0002	0.0002	0.0020	trace	nil	nil	nil	nil
Vulcan American Refined	99.982	0.0016	0.003	0.007	...	0.0035	0.003	...	...	...	...	...
English:												
Chempur	99.9919	0.003	nil	0.0027	0.0006	0.0005	0.0013	nil	trace	...	...	...
Cornish Refined	99.808	0.039	0.018	0.059	0.029	0.033	0.006	0.001	nil	0.005	0.002	nil
Hawthorne Refined	99.891	0.010	0.029	0.028	0.004	0.028	0.010	nil	nil	nil	trace	nil
Mellanear L. & F. (Com.)	99.103	0.227	0.070	0.316	0.087	0.156	0.007	0.009	nil	0.016	0.007	0.002
Mellanear Refined	99.841	0.030	0.016	0.052	0.020	0.025	0.007	0.001	nil	0.006	0.002	nil
Mellanear (Guar. 99.9%)	99.920	0.007	0.023	0.025	0.003	0.012	0.006	nil	nil	nil	0.004	nil
Pass No. 1	99.9894	0.0055	0.001	0.0082	trace	0.0005	0.0028	nil	trace	nil	...	...
Straits:												
E. S. Coy (Penang)	99.904	0.002	0.035	0.028	0.008	0.008	0.008	nil	0.005	...	0.002	nil
Straits Trading (Sing.)	99.895	0.004	0.031	0.029	0.007	0.025	0.009	nil	nil	...	trace	nil
Australian:												
O. T. Lempriere & Co.	99.897	0.018	0.027	0.030	0.002	0.023	0.002	trace	nil	nil	0.001	nil
Pyrmont	99.938	0.017	0.019	trace	nil	0.022	trace	nil	0.004	...	...	...
Dutch:												
Banka	99.9701	0.0022	0.002	0.0128	0.0011	0.0028	0.009	...	...	...	...	...
Billiton	99.934	0.018	0.018	0.019	0.001	0.003	0.007	nil	nil	nil	nil	nil
Dutch L. & F.	99.100	0.289	0.089	0.412	0.014	0.082	0.014	nil	nil	nil	nil	nil
Tulip Refined	99.870	0.041	0.031	0.038	0.003	0.008	0.009	nil	nil	nil	nil	nil
Chinese:												
P.K.M.A.	99.89	0.01	0.02	0.05	0.015	0.005	0.003	0.0005	nil	...	0.001	nil
Y.T.C.	99.17	0.01	0.04	0.61	0.03	0.13	0.003	0.0009	nil	nil	0.001	nil
Belgian:												
Katanga	99.966	nil	0.006	0.012	trace	0.013	0.003	nil	nil	nil	nil	nil

Note:—Except for the first five American Brands, analyses represent pre-war quality.

American Tin Smelter:

Since the last A.S.T.M. review of the tin situation, an important new factor is the Government-owned tin smelting plant which produces the Longhorn brand of tin at Texas City, Tex. Output of tin there since 1942 has totaled 202,821 tons, all of which was produced from imported tin ores, in large part from Bolivia. The quality of this American-made tin is set forth in the typical analyses table (Table II). Whether or not the American tin smelter will continue to operate and to what extent depends upon many factors not now determinable, not the least of which is the continued availability of foreign tin ores. The mine output of the United States and Alaska during the past quarter century has totaled 735 tons of contained tin—about a 3-day supply.

Unless foreign ores are to be imported

TABLE III.—WORLD'S SMELTER PRODUCTION OF TIN.
As estimated by the Bureau of Mines, except as otherwise noted, in tons of 2240 lb.

	1939	1940	1941	1942	1943	1944	1945	1946	1947
Australia	3 294	3 544	3 656	3 024	2 565	2 442	2 400	2 000	2 377
Belgian Congo	2 124	7 832	11 818	13 963	11 068	10 000	10 000	2 372	3 588
Belgium	3 100	[a]	[a]	[a]	[a]	[a]	500	1 405[c]	12 059
China	10 850	13 000	10 000	7 910	4 349	2 160	1 000	1 929	3 600[c]
Germany	7 000	3 000	3 000	4 000	5 000	4 000	[a]	...	...
Japan	2 000	2 500	[a]	4 000	[a]	2 000	1 000	68	600[c]
Malaya	81 536	126 945	125 000	25 000[b]	25 000[b]	25 000[b]	5 000[b]	7 500	29 318
Netherlands	14 600	2 967	[a]	[a]	[a]	[a]	[a]	945[c]	8 750
Netherlands East Indies	13 941	22 035	23 000	8 000	12 000	3 000	500	...	...
Siam	[a]	[a]	[a]	[a]	[a]	[a]	7 000		...
United Kingdom	37 400	42 000[b]	40 000	30 000	31 000	25 000	28 000	28 500	27 544
United States		1 391	1 839	16 198	21 489	30 884	40 475	43 500	33 280
Sundry others	2 000	2 500	3 000	4 500	4 500	4 500	3 500	5 672	2 361[c]
Totals	177 600	227 000	225 000	120 000	125 000[b]	114 000[b]	100 000[b]	93 900	123 500

[a] Included in the total.
[b] As estimated by the American Metal Market.
[c] Production estimated by International Tin Study Group.

TABLE IV.—CONSUMPTION OF PRIMARY AND SECONDARY TIN IN THE UNITED STATES, BY FINISHED PRODUCTS (TIN CONTENT), FOR SELECTED PERIODS DURING 1937-1947.
Sources: 1937-1947, Bureau of Mines, U. S. Department of Interior; 1946, U. S. Department of Commerce.

	1937–1939 Average		1942–1944 Average		1945		1946		1947	
	Long tons	Per cent of total	Long tons	Per cent of total	Long tons	Per cent of total	Long tons	Per cent of total	Long tons	Per cent of total
Metallic:										
Tinplate	33 136	42.8	25 072	29.4	26 080	31.2	27 051	33.7	31 833	36.5
Solder	16 645	21.5	13 409	15.7	14 329	17.1	17 255	21.5	19 696	22.6
Bronze and brass	5 621	7.3	30 168	35.4	27 065	32.4	22 305	27.8	21 148	24.3
Babbitt	5 459	7.0	7 546	8.8	7 828	9.4	6 833	8.5	6 829	7.8
Collapsible tubes	3 502	4.5	738	0.9	559	0.7	898	1.1	1 008	1.2
Tinning	2 254	2.9	3 057	3.6	2 592	3.1	2 230	2.8	2 825	3.2
Foil	1 915	2.5	422	0.5	245	0.3	238	0.3	356	0.4
Terne plate	1 286	1.7	685	0.8	741	0.9	[a]	[a]	[a]	[a]
Type metal	1 204	1.6	1 195	1.4	1 291	1.5	1 378	1.7	1 372	1.6
Pipe and tubing	950	1.2	208	0.2	293	0.3	324	0.4	479	0.5
Bar tin	945	1.2	981	1.1	1 094	1.3	[b]	[b]	[b]	[b]
Galvanizing	939	1.2	28	[c]	...	...	[b]	[b]	[b]	[b]
White metal	450	0.6	163	0.2	216	0.3	[b]	[b]	[b]	[b]
Miscellaneous alloys	404	0.5	955	1.1	606	0.7	[b]	[b]	[b]	[b]
Nonmetallic:										
Tin oxide	1 068	1.4	44	0.1	...	...	[d]	[d]	[d]	[d]
Other chemicals	1 011	1.3	317	0.4	644	0.8	118	0.2	173	0.2
Other	655	0.8	341	0.4	[d]	[d]	1 604	2.0	1 462	1.7
Total	77 444	100.0	85 329	100.0	83 583	100.0	80 234	100.0	87 181	100.0

[a] Included with tin plate data.
[b] Included in "other."
[c] Less than 0.05 per cent.
[d] Included in "other chemicals."

and smelted competitively, then there will arise the question as to whether a Governmental subsidy may be justified on the ground that the Texas City smelter is an essential defense plant, to be kept in full or partial operation or in a standby condition.

Secondary Tin:

During the seven prewar years, consumption of secondary tin in the United States averaged 15,000 tons per annum, about 20 per cent of the total tin consumption. Wartime conservation raised this to 42 per cent. Such recovery is in two general categories (1) at detinning plants, employing electrolytic and chemical separation means, and (2) in the combination of various forms of tin-bearing scrap to produce alloys for use in bearing metals, bronzes, and solders. The bulk of this secondary recovery goes to bronze, babbitt, solder, and chemicals.

Consumption:

The United States will no doubt continue to absorb the largest part of the world's tin output—almost as much as all the rest of the tin-consuming nations put together. However, while tin continues in short supply, intergovernmental allocation will probably remain in effect.

The effects of war-time conservation controls will be noted in Table IV, which shows the uses of tin in the United States during the past ten years.

Government Control:

That Governmental controls of tin are to be continued is indicated by the following January, 1948, report to Congress by the Secretary of Commerce:

> "In order to ensure an equitable distribution of world tin supplies, international allocations of tin metal are made through the mechanism of the Combined Tin Committee, an intergovernmental body of which the United States is a member. Current allocations to the United States in line with our pre-war share of total consumption permit the fulfillment of less than three-fourths of domestic requirements for primary tin. Tin distribution controls are needed, therefore, in order to direct the limited amounts available in the most equitable manner.
>
> "The restrictions have been liberalized as rapidly as conditions have warranted. The permissible tin content of foil, collapsible tubes, solders, babbitts, and bronzes has been increased, and the use of tin in limited quantities has been granted for a few new applications. However, some uses are still prohibited. These include toys, jewelry, ornaments, novelties, art objects, numerous plating operations, and the use of certain tin chemicals as opacifiers, stabilizers, and reducing agents. The manufacture and use of cans made of tin plate and terne plate are controlled. Cans made exclusively of black plate or tin plate waste, terne plate waste, tin plate waste waste, or terne plate waste waste are not restricted by the order. The general intent of the order is to provide an adequate container for packaging various commodities.
>
> "Future problems center about the transition from a period of control to a return of private distribution and unlimited use. These problems are both domestic and international. Except for small amounts in a few limited areas, there is today no place where tin can be purchased by private individuals. Major sources of supply are under the control of foreign governments, who are willing to sell only in bulk. Until other arrangements are made, the United States Government must continue to function as practically the sole importer of this metal."

REPORT OF COMMITTEE B-3*

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in Washington, D. C. on March 3, 1948.

The committee consists of 105 members, of whom 86 are voting members; 29 are classified as consumers, 39 as producers, and 18 as general interest members.

The election of officers for the ensuing two years resulted in the selection of the following:

Chairman, K. G. Compton.
Vice-Chairman, Wm. H. Finkeldey.
Secretary, A. W. Tracy.

At its meeting on March 3, 1948, the committee voted to continue as tentative without revision the following three methods of test under its jurisdiction:

Tentative Methods of Test for:

Total Immersion Corrosion Testing (B 185 - 43 T),
Salt Spray (Fog) Testing (B 117 - 44 T), and
Alternate Immersion Corrosion Testing (B 192 - 44 T).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman)—The procedures described in the Tentative Method of Test for Total Immersion Corrosion Testing (B 185 - 43 T) will be further investigated for ability to give reproducible results. Several laboratories will make tests on copper, lead, and nickel in normal sulfuric acid; copper and lead in 5 *N* sodium hydroxide; and copper or red brass, lead and prime western zinc in 1 *N* sodium chloride. Tests will be made with specimens moving at 5 ft. per min. and also at 15 ft. per min.

Subcommittee III on Spray Testing (C. E. Heussner, chairman)—A subgroup has been appointed to review the work on factors affecting the Tentative Method of Test for Salt Spray (Fog) testing (B 117 - 44 T) and to recommend any necessary revisions. The work done on some of these factors is reported in a paper, "Apparatus and Factors in Salt Fog Testing," by V. M. Darsey and W. R. Cavanagh, appended hereto.[1a]

Subcommittee III will work toward the development and standardization of an acetic acid - salt spray test which has been reported to have special merit in evaluating the quality of plated coatings on zinc-base die castings.

Subcommittee IV on Humidity Test (L. J. Waldron, chairman) has been authorized to do the necessary work to standardize a humidity test of general applicability. To this end, A.S.T.M. Committees D-14 on Adhesives, D-19 on Industrial Water, D-20 on Plastics, and C-19 on Structural Sandwich Materials have been invited to appoint representatives to this committee.

Subcommittee VI on Atmospheric Corrosion W. H. Finkeldey, chairman).—A tentative outline of atmospheric tests on magnesium alloys, to be carried out jointly by this subcommittee and Committee B-7 on Light Metals and Alloys, has been drawn up and submitted to Committee B-7 for study.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[1a] See p. 153.

Subcommittee VII on Weather (F. L. LaQue, chairman)—A subgroup has been appointed to study the availability and usefulness of instruments for measuring air pollution. Another small group has been appointed to study the relative corrosivity at the various A.S.T.M. test sites and to obtain data by exposing a large number of zinc and iron specimens.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman)—A progress report on the five year-atmospheric exposure tests on stainless[2] steel coupled to other metals is appended to this report.[3]

The atmospheric exposure test program of magnesium alloys coupled to other metals has progressed to the point where disk couples and couples formed by winding wire on threaded bolt materials will be exposed this summer.

This report has been submitted to letter ballot of the committee which consists of 86 voting members; 64 returned their ballots; of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. S. RAWDON,
Chairman.

A. W. TRACY,
Secretary.

---

[2] The word "stainless" is employed in this report as applying to austenitic chromium-nickel alloy steels. The term used in A.S.T.M. specifications for this material is "corrosion-resistant steel."

[3] See p. 167.

APPENDIX

APPARATUS AND FACTORS IN SALT FOG TESTING*

By V. M. Darsey[1] and W. R. Cavanagh[1]

Adoption by the Society in 1944 of a procedure specifying the conditions of the salt fog environment inside the test chamber, its calibration **(1)**[2] and maintenance, has contributed greatly to the uniformity and reproducible results obtained with the salt spray fog testing method B 117 - 44 T.[3] Standardizing on a *single* salt fog test has resulted in a concentration of the research work on the method which has resulted in a better understanding of the important factors involved. It is recognized that a dry salt fog produces less corrosion on exposed specimens in the test than a wet fog. A procedure for producing a uniform and salt fog in the test chamber and its measurement is a requirement of B 117 - 44 T.

By following this procedure, it is possible to investigate some of the important factors of the test with the knowledge that variations in the salt concentration, collection rate of salt fog particles and pH of the collected solution can be detected. It is recognized that additional research on the test is advisable, especially in the use of the method for testing non-ferrous metal and alloys.

Some of the work discussed by Subcommittee III of Committee B-3 at its February, 1946, meeting in Pittsburgh, indicated that additional information was desirable on the following important factors of salt spray fog testing:

(*a*) Effect of temperature on pH of salt solution,

(*b*) Volume and pressure of air used to atomize salt solution,

(*c*) Effect of salt fog collection rate on corrosion,

(*d*) Effect of temperature and specimen position on rate of corrosion,

(*e*) Design of salt fog testing apparatus, and

(*f*) Corrosion of non-ferrous metals, nickel, zinc, cadmium, aluminum, and magnesium in test.

Considerable research work on these factors except the latter has been conducted by the authors and is presented herewith in the hope it will aid in further improving the test method and stimulating worker interest.

A Measure of Corrosion

To evaluate the effect of such factors as pH, rate of fog settling, air pressure, temperature, and specimen position in the salt fog test, it was first necessary to establish a means of measuring corrosion. For the purpose of this investigation, the weight loss in mg. per sq. cm. of a steel panel when exposed to the salt fog for 24 hr. is used as a measure of its corrosiveness. The steel panels used for this purpose conform to the requirements of A.S.T.M. Method for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 45 T).[4] These panels had a Rockwell hardness of 55 to 65 and a finish equivalent to a No. 5 (dull) finish as produced by Carnegie-Illinois Steel Corp. The panels were cleaned by washing in mineral spirits followed by degreasing in trichlorethylene vapors. After cleaning, the panels were weighed and the backside coated with a heavy rust preventive to protect it from the salt fog. The panels thus prepared were exposed for 24 hr. at 15 deg. off the vertical in the salt fog chamber with the uncoated side up. At the end of the exposure period, the panels were water-rinsed, dried, and vapor-degreased to remove the rust preventive material. The corrosion products formed on the panels by action of the salt fog were removed by immersing the panels in a stripping solution consisting of 100 parts by weight of concentrated hydrochloric acid, two parts of antimony trioxide, and five parts of stannous chloride (2). The panels were then water rinsed, dried, and re-weighed. The weight loss per square centimeter was calculated and this figure used to represent the corrosiveness of the salt fog.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] President and Research Chemist, respectively, Parker Rust Proof Co., Detroit, Mich.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 166.

[3] "Tentative Method of Preparation of Salt Spray Fog Testing," 1946 Book of A.S.T.M. Standards, Part I-B, p. 773.

The nozzles for atomizing the salt solution were non-adjustable siphon type, external mixing, with round spray pattern and of all Monel metal construction as supplied by Spraying Systems, Inc., Chicago, Ill. Four air nozzles, Nos. 60, 64, 70, and 64–5 were used in the experiments. The first three had inside diameters of 0.060, 0.064, and 0.070 in., respectively, and the fourth had an inside diameter of 0.064 in., but rests 0.005 in. below the tip of the fluid nozzle rather than flush as do the others. Three fluid nozzles, Nos. 1650, 2050, and 2850 were used. The outside diameters of the fluid nozzles were 0.050 in. and the inside diameters were 0.016, 0.020, and 0.028 in., respectively. Any of the four air nozzles may be used with any of the fluid nozzles as shown in Fig. 1.

The salt fogs obtained inside the chambers with the separate nozzles were checked according to the procedure outlined in B 117 - 44 T. The settling rate was determined by collecting a sample of the fog in a 10-cm. diameter funnel inserted through a rubber stopper into a 100-ml. graduated cylinder and expressed as milliliters collected per hour. The collected fog solution was checked for pH colorimetrically and the salt content calculated from its chloride content as determined by titrating an aliquot with 0.1 *N* silver nitrate.

The sodium chloride used in preparing the 20 per cent salt solution was the Morton Salt Co. Butter Flake Grade which conforms to the requirements of Method B 117 - 44T. The salt solution was prepared with Detroit City water and adjusted to the desired pH by additions of C.P. hydrochloric acid.

TYPICAL ANALYSIS OF DETROIT CITY TAP WATER.

Turbidity	0.5
Total solids	114.0
Iron (Fe), ppm	0.020
Silica (SiO_2), ppm	0.5
Al_2O_3 and Fe_2O_3 (R_2O_3), ppm	2.8
Sulfate, (SO_4), ppm	12.8
Chloride (Cl), ppm	8.0
Calcium (Ca), ppm	26.8
Magnesium (Mg), ppm	7.1
Sodium (Na), ppm.[a]	3.2
Total Hardness ($CaCO_3$), ppm	96.1
Total Alkalinity ($CaCO_3$), ppm	78.0
CO_3 Alkalinity ($CaCO_3$), ppm	0
HCO_3 Alkalinity ($CaCO_3$), ppm	78.0
Free CO_2 ($CaCO_3$), ppm	2.0
Non CO_3 Hardness ($CaCO_3$), ppm	18.1
Oxygen consumed, ppm	1.0
Dissolved Oxygen, ppm	14.0
Nitrogen Organic, ppm	0.550
Nitrogen Albuminoid, ppm	0.128
Nitrogen Ammonia, ppm	0.040
Nitrogen Nitrite, ppm	0.002
Nitrogen Nitrate, ppm	0.150
pH	7.7
Temperature, deg. Fahr	37.0

[a] By difference.

All pH measurements were made with a Hellige comparator using standard disks and the indicators shown in Table I.

---

[4] "Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products," 1946 Book of A.S.T.M. Standards, Part II, p. 1601.

EFFECT OF TEMPERATURE ON pH OF SALT SOLUTION

Salt solutions ranging in pH from 6.8 to 7.2 when contained in a supply tank at room temperature outside the fog chamber were observed to range in pH from 8.4 to 8.7 when atomized and collected inside the fog chamber at 95 F. In addition, salt solution removed from the reservoir tank inside the fog chamber which had not been atomized but maintained at 95 F. indicated a similar increase in pH to 8.3 to 8.7.

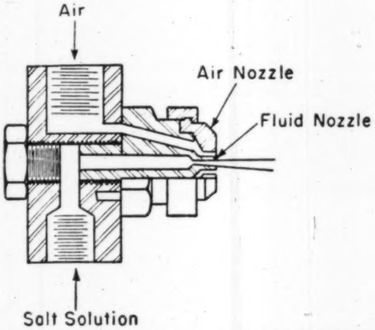


FIG. 1.—Cross Section of Monel Metal Nozzle Used to Atomize Salt Solution.

To account for the increase in pH of a salt solution maintained at a temperature of 95 F., a glass stoppered flask was filled with a 20 per cent sodium chloride solution having a pH of 6.9 and placed in the fog chamber for 40 hr. At the end of this period the pH of the salt solution in the flask was measured along with the solution in the reservoir inside the chamber and the fog collected during this time. The pH of all the samples was found to be the same, 8.7.

These differences in pH are analogous to the difference in the pH of distilled water which has been saturated with air containing carbon dioxide (pH 5.7) and the theoretical value (pH 7.0). This difference has been attributed to the presence of 0.03 per cent by volume of carbon dioxide in the water (**3**).

To determine whether these differences result from the loss of dissolved carbon dioxide as the solution is warmed to 95 F. in the salt fog chamber, two solutions of 20 per cent sodium chloride were prepared—the first sample with freshly boiled tap water and the second with unheated tap water. The pH of the two samples as prepared was 8.4 and 7.2, respectively. Both samples were heated to boiling for one minute and cooled rapidly to room temperature. The pH of the two samples was then found to be 8.7 and 9.1, respectively. The fact that the pH of the sample prepared from boiled water rose only 0.3 while that of the sample prepared from unheated water rose 1.9 indicated the pH will remain fairly constant if the carbon dioxide is removed from the water before preparing the salt solution.

TABLE I.—INDICATORS USED TO DETERMINE pH VALUES USING STANDARD COLOR DISKS WITH A HELLIGE COMPARATOR.

Indicator	pH Range
Methyl red	4.4 to 6.0
Bromthymol blue	6.0 to 7.6
Cresol red	7.2 to 8.8
Cresol phthalein	8.4 to 9.8

The procedure was considered of adjusting the salt solution to a pH below that required in Method B 117 - 44T in order that the specified pH would be obtained when the solution was atomized at 95 F. This procedure was abandoned in view of the variation of carbon dioxide content of water at different temperatures and at different locations. It was noted, however, that samples of salt solution which are boiled gently for 30 sec. and cooled to room temperature immediately before checking gave pH values very close to those obtained if the solution was maintained at 95 F. for 24 hr. or more.

Due to the effect of temperature on the pH of the salt solution, the requirement for the pH of such solution in Method B 117 - 44T should be based on the collected fog rather than the salt solution. The specified pH of the collected fog will be obtained if the salt solution is allowed to stand for 48 hr. at approximately 95 F. before the pH is adjusted. When it is desired to adjust the pH of a salt solution at room temperature, it is necessary to remove a sample and boil gently 30 sec. before determining the pH. From this information the pH of the salt solution can be adjusted by adding acid or alkali, as indicated.

EFFECT OF MATERIALS ON pH OF SALT SOLUTION

A salt solution which had a pH of 7.0 after being gently boiled for 30 sec. was placed in a Monel metal storage tank connected to the salt fog test chamber. This solution was used for operating the salt fog chamber and a sample of the collected fog was found to test above the 6.5 to 7.2 pH range of Method B 117 - 44 T. Investigation revealed that the pH of the salt solution in the Monel metal storage tank had changed until a boiled sample now had a pH of 8.6.

A strip of Monel metal was placed in a glass-stoppered flask which was then filled with 20 per cent salt solution, a sample of which had a pH of 6.5 after being gently boiled for 30 sec. A second glass flask was filled with the same solution as a control. Both flasks were allowed to stand 48 hr. at room temperature after which the pH of each solution was determined on a boiled sample. The solution in contact with the Monel metal had a pH of 8.0 while that of the control remained 6.5. This experiment clearly indicates the effect on the pH of the salt solution in contact with Monel metal.

To find a satisfactory material for storing the salt solution, various materials were checked in a similar manner for their effect on the pH of salt solutions. Monel, nickel, welded stainless steel, phenolic varnish-coated steel, Koroseal[5]-coated steel, and Saran[6] tubing were placed in separate flasks and covered with 20 per cent salt solution, the pH of which ranged from 5.0 to 5.4 at room temperature. The pH of the salt solution was also checked on samples which had been gently boiled for 30 sec. before being placed in the flask and after it had been in contact with the test piece for 48 hr. The results are recorded in Table II.

The use of either phenolic varnish-coated steel or Koroseal-coated steel provides a satisfactory container for storing at room temperature a salt solution which has been adjusted to produce a salt fog having a pH of 6.5 to 7.2 when atomized at 95 F.

TABLE II.—EFFECT OF VARIOUS MATERIALS ON THE pH OF 20 PER CENT SODIUM CHLORIDE.

Material	pH Room Temperature	pH at Start[a]	pH After 48 hr.[a]
Monel	...	6.5	8.0
Nickel	5.0	6.9	8.3
Welded stainless steel	5.0	6.9	8.0
Phenolic varnish-coated steel	5.2	6.2	6.2
Koroseal-coated steel	5.4	6.8	6.8
Saran tubing	5.4	6.8	6.8

[a] pH was checked on samples which had been gently boiled for 30 sec.

In Table VII are shown the results obtained when a salt solution having a pH of 6.5 to 7.2 after gently boiling for 30 sec. is stored at room temperature in a Koroseal-lined steel tank until atomized at 95 F.

EFFECT OF VOLUME AND PRESSURE OF AIR USED TO ATOMIZE SALT SOLUTION

In view of the variety of nozzles and air pressures in use and permitted in Method B 117 - 44T, experiments were conducted to determine the effect of the

---

[5] B. F. Goodrich Co.

[6] Dow Chemical Co.

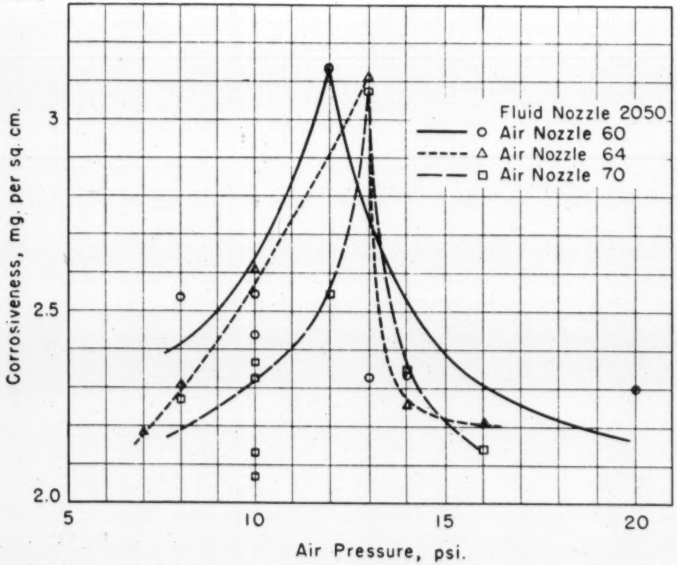


FIG. 2.—Effect of Different Nozzles and Air Pressures with Air Pressures Varying ±0.5 psi. Using Fluid Nozzle 2050 and Air Nozzles 60, 64, and 70.

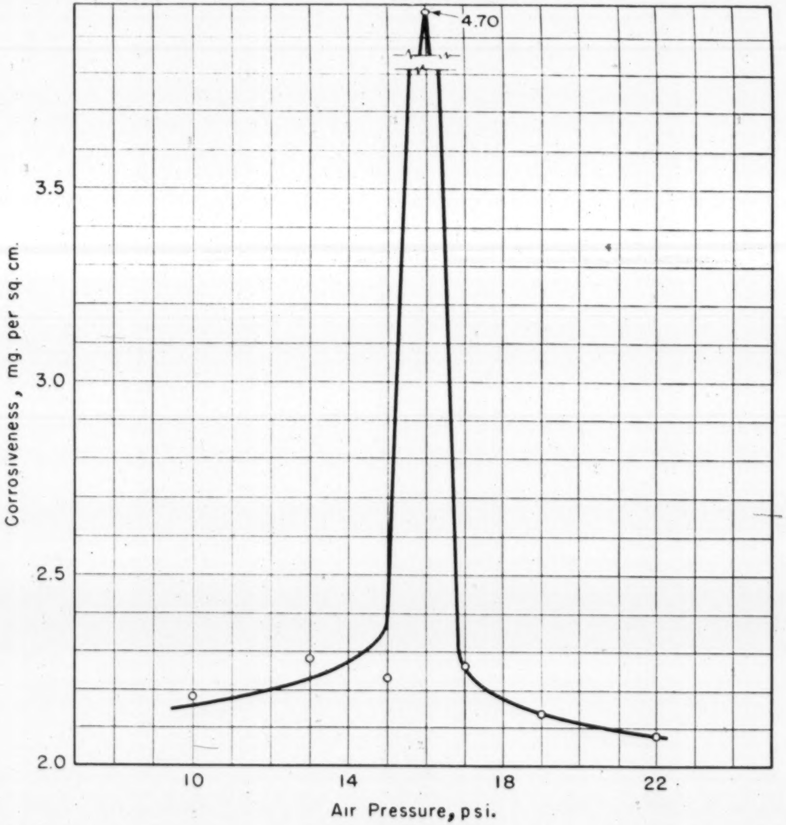


FIG. 3.—Effect of Different Air Pressures Varying ±0.5 psi. Using Fluid Nozzle 2850 and Air Nozzle 64-65.

volume of air, the pressure of the air, and the size of the nozzle on the corrosiveness of the salt fog.

The corrosiveness of fogs produced at various pressures was determined using air nozzles Nos. 60, 64, and 70 with fluid nozzle No. 2050 and also using air nozzle No. 64–5 with fluid nozzle No. 2850. A series of determinations was run for each nozzle combination at pressures of from 4 to 20 psi. except for the last combination in which case pressures of from 10 to 25 psi. were used. The air pressures in these tests were measured by a low-pessure DeVilbiss gage.

TABLE III.—EFFECT OF DIFFERENT NOZZLES AND AIR PRESSURES WITH AIR PRESSURES VARYING ± 0.5 PSI.

	Pressure, psi.	Fog Collection Rate, ml. per hr.	NaCl in Fog, per cent	Corrosiveness, mg. per sq. cm.
Fluid nozzle No. 2050 and air nozzle 60	10	1.1	15.2	2.44
	10	2.2	17.2	2.55
	20	1.2	17.0	2.30
	12	0.8	18.1	3.14
	8	0.7	16.6	2.54
	13	0.8	18.1	2.33
	14	0.9	18.1	2.34
Fluid nozzle No. 2050 and air nozzle 64	10	0.9	15.9	2.61
	9	1.2	17.2	2.41
	8	1.0	18.1	2.31
	7	0.8	18.2	2.19
	13	1.2	17.0	3.12
	14	1.0	16.8	2.26
	16	0.9	17.7	2.21
Fluid nozzle No. 2050 and air nozzle 70	10	0.9	16.0	2.07
	10	3.1	18.5	2.13
	10	3.0	18.2	2.33
	10	1.0	15.2	2.37
	8	1.0	18.1	2.27
	12	1.0	18.0	2.55
	16	0.9	18.0	2.14
	14	1.0	17.3	2.34
	13	0.8	17.8	3.08
	10	0.8	17.1	2.56
Fluid nozzle No. 2850 and air nozzle 64-5	10	1.2	19.4	2.18
	13	1.3	19.0	2.29
	19	1.5	20.1	2.14
	22	1.5	19.6	2.08
	17	1.5	19.3	2.26
	15	1.5	19.2	2.23
	16	1.4	18.8	4.70

The results obtained are shown in Table III. These results are shown graphically in Figs. 2 and 3.

The corrosiveness of the salt fogs produced with the No. 2050 fluid nozzle shows a peak at 12 or 13 psi. regardless of which air nozzle was used. A similar peak was noted with the No. 2850 fluid nozzle at 16 psi.

To investigate the points of maximum corrosiveness noted, a mercury manometer was substituted for the DeVilbiss pressure gage in order to determine more closely the pressure of the air. The manometer indicated that the air pressure at the nozzle was varying ± 0.5 psi. This variation was a result of the line pressure varying between 60 and 80 psi. at the compressor. The compressor was observed to be running for one minute and off for four minutes with a corresponding five-minute cycle at the

TABLE IV.—EFFECT OF DIFFERENT AIR PRESSURES VARYING ± 0.1 PSI.

	Nozzle Pressure, psi.	Fog Density, ml. perhr.	NaCl in Fog, per cent	Corrosiveness, mg. per sq. cm.
Fluid nozzle 2050 and air nozzle 64-5	12.4	1.0	17.4	2.36
	12.6 to 12.7	0.7	15.3	2.41
	12.7 to 12.9	1.4	18.0	2.38
	12.8 to 13.1	1.3	17.9	2.21
	12.4 to 12.6	1.5	16.7	2.13
	12.6	1.2	17.2	2.21
	13.5 to 13.6	1.4	18.2	2.19
	13.3	0.6	15.9	2.37
	13.2 to 13.3	1.4	17.5	2.13
	12.7 to 12.8	1.3	18.0	2.27
	Average........	1.2	17.2	2.27
	Deviation maximum........	+0.3 −0.6	+0.8 −1.9	+0.14 −0.14
	Average deviation..........	±0.24	±0.75	±0.091
Fluid nozzle 2050 with air nozzle 64	11.1 to 11.2	1.3	17.6	2.19
	11.6 to 11.7	1.5	17.4	2.19
	11.9	1.5	17.8	2.22
	12.2 to 12.3	1.5	17.6	2.20
	12.4	1.6	17.3	2.25
	12.6 to 12.7	1.6	17.4	2.15
	14.0	1.4	18.1	2.21
	13.6 to 13.7	1.6	17.9	2.12
	13.9 to 14.0	1.3	17.2	2.12
	13.9 to 14.0	1.2	17.5	2.19
	14.2 to 14.3	1.0	17.6	2.24
	14.1 to 14.3	1.0	16.7	2.27
	14.1 to 14.3	1.2	17.5	2.16
	14.1 to 14.3	1.2	17.9	2.22
	14.4 to 14.5	1.2	17.8	2.17
	14.6 to 14.7	1.3	18.1	2.14
	Average........	1.3	17.6	2.19
	Deviation maximum........	+0.3 −0.3	+0.5 −0.9	+0.08 −0.07
	Average deviation	±0.16	±0.26	±0.035

nozzle. The manometer also indicated that a gage reading of 12 psi. was actually 13.5 psi. at the nozzle.

By installing a second diaphragm-type reducing valve in the line in series with the original reducing valve, the variation in air pressure was reduced to ±0.1 psi.

Additional tests were run using the more closely controlled air pressures. The air nozzle No. 64 and the fluid nozzle No. 2050 were used in those tests and the fog collection rate was varied by changing the distance between the nozzle and a baffle. Collection rates of 0.4 and 1.3 ml. per hr. were used at air pressures of 12 to 13 psi. by the gage. When the air pressures were kept constant to within 0.1 psi., the points of maximum corrosiveness previously noted were not evident. The results of these tests are shown in Table IV.

TABLE V.—EFFECT OF COLLECTION RATE ON CORROSIVENESS OF SALT FOG.

Collection Rate, ml. per hr.	Salt Concentration of Fog, per cent	Corrosiveness, mg. per sq. cm.		
		First panel	Second panel	Average
0.13	18.5	2.44	2.78	2.61
0.17	14.6	2.96	3.23	3.09
0.33	16.2	2.56	2.57	2.56
0.33	14.1	2.57	2.60	2.58
0.41	16.1	2.44	2.48	2.46
0.75	15.7	2.80	2.98	2.89
0.88	15.4	2.58	2.68	2.63
1.1	16.3	2.44	2.60	2.52
1.2	17.1	2.44	2.51	2.48
2.3	17.6	2.25	2.27	2.26
2.6	17.6	2.20	2.25	2.22
3.3	18.3	2.17	2.15	2.16
4.6	18.4	2.10	2.10	2.10

It appears that the points of maximum corrosiveness noted above are associated with the fluctuations in the air pressure at a critical pressure and that the critical pressure is dependent upon the size of the fluid orifice and independent of the size of the air orifice.[7] This possibility of increased corrosiveness can be avoided by operating above or below the critical pressure provided the critical pressure is known for a particular nozzle, or by controlling the air pressure to within ±0.1 psi.

EFFECT OF SALT FOG COLLECTION RATE ON CORROSION

The limits for the fog collection rate required in method B 117-44T are 0.5 to 3.0 ml. per hr., but little evidence is available as to the effect of different collection rates on the corrosiveness of the salt fog. Experiments were conducted to determine the relationship between the collection rate of the fog and corrosion. The two greatest collection rates (3.3 and 4.6 ml. per hr.) were obtained by using fluid nozzle No. 2850 and air nozzle No. 64. The remaining tests were run using fluid nozzle No. 2050 and

TABLE VI.—EFFECT OF COLLECTION RATE ON CORROSIVENESS OF SALT FOG.

Collection Rate, ml. per hr.	Salt Concentration of Fog, per cent	Corrosiveness, mg. per sq. cm		
		First panel	Second panel	Average
0.1	16.8	1.84	2.09	1.97
0.2	...	1.89	1.86	1.88
0.3	17.2	2.02	2.09	2.06
0.3	20.1	1.95	2.00	1.97
0.3	19.6	2.35	1.88	2.11
0.4	20.3	2.00	1.95	1.97
0.4	19.2	1.96	1.97	1.97
0.4	20.6	2.02	1.92	1.97
0.4	19.5	2.15	1.96	2.04
0.4	18.5	2.09	1.98	2.04
0.4	18.9	2.00	1.98	1.99
0.5	19.4	1.80	1.93	1.87
0.5	18.6	1.80	1.96	1.88
0.6	17.6	2.01	2.07	2.04
0.6	...	2.10	2.10	2.10
0.6	19.6	2.15	2.16	2.16
0.7	19.4	1.84	1.86	1.85
0.7	18.3	1.96	1.90	1.93
0.8	20.4	1.88	1.87	1.88
0.8	20.0	2.06	2.08	2.07
0.8	19.5	2.04	2.13	2.09
0.8	20.2	1.98	1.85	1.91
1.0	20.0	2.35	2.19	2.27
1.3	19.0	2.27	2.21	2.24
1.5	19.6	2.11	2.17	2.14
1.6	19.0	1.99	1.94	1.97
1.6	19.6	2.28	2.17	2.23
1.8	19.7	1.89	2.05	1.97
1.9	19.5	2.02	2.23	2.13
3.0	20.3	2.12	2.15	2.13
3.3	20.1	2.20	2.23	2.21
4.+	19.9	1.92	2.18	2.05

---

[7] The effect of fluctuating air pressure was also noted in the salt fog cabinet at the Spring Division of the Borg-Warnet Corp., Bellwood, Ill. The corrosiveness of this cabinet was more than doubled when the air pressure was varying between 10.8 and 14.1 psi. over what it was when the pressure was varying between 11.9 and 14.9. The nozzle used was a Spraying Systems No. 2050 fluid nozzle and a No. 64 air nozzle.

air nozzle No. 64 and controlling the collection rate by varying the distance between the nozzle and a baffle. The results of these tests are shown in Table V.

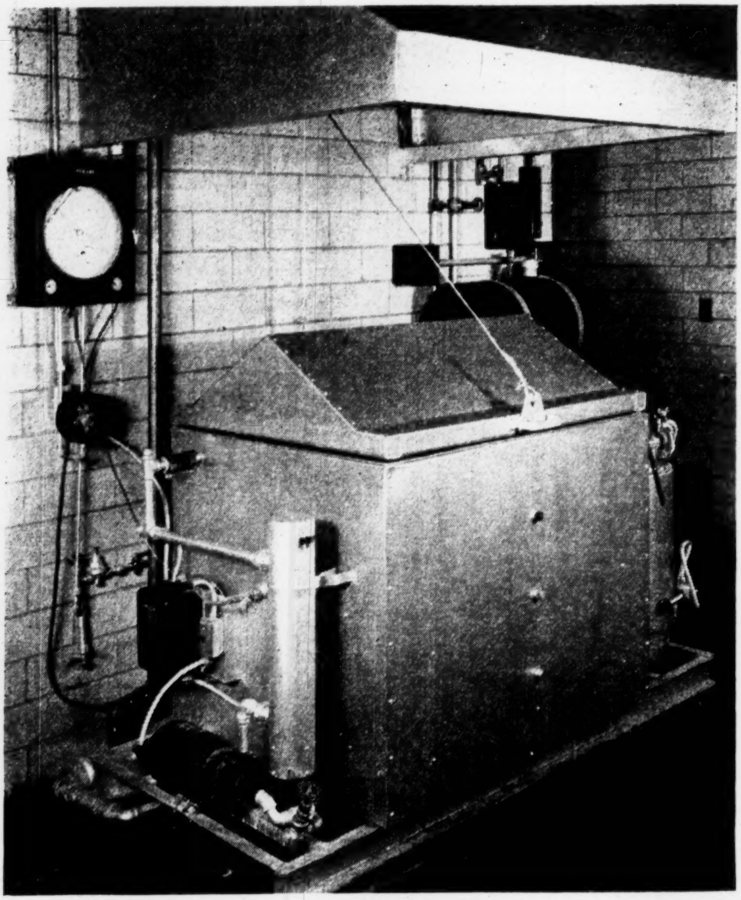


FIG. 4.—Salt Fog Testing Cabinet.

The corrosiveness of the fog increased as the collection rate and salt concentration decreased. This increase in the corrosiveness may be due to either the decrease in the salt concentration in the fog or the decrease in the collection rate.[8]

To determine the effect of variations in the collection rate with little or no change in the salt concentration of the fog, the above tests were repeated in the 25-cu. ft. salt fog cabinet which is described later in this paper. These tests were run using air nozzle No. 64-5 and fluid nozzles Nos. 1650, 2050, and 2830. The results are shown in Table VI.

There appears to be a slight increase in the corrosivity as the collection rate increases, but the increase does not appear to be significant in view of the variation obtained at the same collection rate.

Variations in the collection rate within the limits of Method B 117 - 44 T do not affect the corrosiveness of the salt fog.

---

[8] Unpublished work of the authors has indicated that salt fogs below 20 per cent salt concentration are more corrosive (as measured by steel panels) than salt fogs of a higher salt concentration.

The increase in the corrosiveness observed previously at low collection rates is attributed to the low salt concentration obtained.

DESIGN OF SALT FOG CABINET

Because of the difference in corrosion rate experienced in the operation of salt fog testing equipment at room temperature, a temperature of 95 F. $^{+2}_{-3}$ F. was made a requirement of the test method. It was believed easier to maintain this temperature which represents about the highest experienced in summer than to provide cooling facilities to maintain the spray chamber at the temperature usually experienced from the operation at room temperature in the winter months.

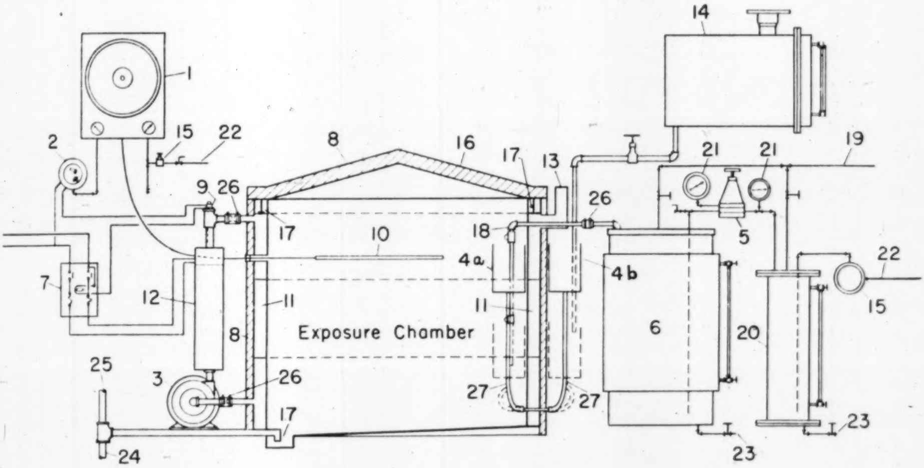


FIG. 5.—Diagram of Salt Fog Testing Cabinet.

1. Temperature recorder controller for fog chamber. 2. Pneumatic switch. 3. Pump for continuously circulating water. 4. Adjustable constant level salt reservoirs. a. Monel metal. b. Rubber lined. 5. Air pressure regulator (nullmatic). 6. Saturator tower—thermostatically controlled. 7. Relay switch for heater. 8. Insulation. 9. Water limit thermostat. 10. Bulb for recorder controller. 11. Jacket for circulating water. 12. Water heater. 13. Vent on water jacket. 14. Rubber lined salt reservoir. 15. Reducing valve. 16. Hinged cover. 17. Water seal. 18. Monel nozzle. 19. Water line. 20. Water scrubber. 21. Pressure gage. 22. Air line. 23. Drain. 24. To sewer. 25. Vent. 26. Rubber coupling. 27. Rubber tubing.

The maintenance of uniform temperature in the salt spray chamber as required in Method B 117-44T is somewhat more difficult than generally believed. Several methods have been proposed for maintaining the desired temperature in such equipment, the principal ones designated as follows:

1. Provide a large room maintained at a temperature of approximately 95 F. in which is located the salt fog testing apparatus. This scheme while capable of maintaining satisfactory temperatures has the disadvantage of being inconvenient at the time of inspecting and rating of specimens tested in the spray chamber.

2. Provide a water seal around the spray chamber in which the temperature is adjusted to maintain the inside of the chamber at 95 $^{+2}_{-3}$ F.

3. Provide for an air jacket around the fog chamber in which the temperature can be regulated to maintain the inside at the desired temperature.

In maintaining the temperature of the salt fog chamber, it is necessary to prevent overheating the cabinet at any one location or else moisture will be evaporated from the salt fog particles and salt deposited at such areas. Such equipment makes it difficult to maintain the collected fog solution at the desired salt concentration.

Realizing the various factors involved in salt fog testing cabinets, the authors designed and had built the apparatus shown in Figs. 4 and 5. The exposure chamber is constructed of Monel metal with a water jacket around the four sides. The top is in the shape of an inverted V and is designed to permit any drops of salt solution to fall about $\frac{1}{4}$ in. from the sides and not on the test specimens or into the water seal at the top of the chamber. The fog is vented through a water seal located at the bottom of the exposure chamber.

The temperature is maintained by a continuous circulation of warm water through the water jacket. A pump is used to circulate the water, the temperature of which is maintained by a heater mounted on the discharge side of the pump. Both the pump and the heater are constructed of mild steel and are connected to the Monel jacket by rubber couplings. The temperature is controlled and recorded by an air-operated recorder controller, the bulb being located in the exposure chamber. To prevent overheating the water when the box is open and thus overheating the chamber after closing, a thermostat is mounted at the exit end of the heater which turns off the heater if the water exceeds 97 F.

The salt solution is stored in a Koroseal-lined tank which is sealed except for the discharge line. The salt solution is atomized by a Monel metal nozzle (Spraying Systems Co. Pneumatic Atomizing Nozzle set up No. 3-B using fluid nozzle No. 1650 and air nozzle No. 64–5) which can be located at any desired height in the chamber. The No. 1650 fluid nozzle is used rather than No. 2050 or No. 2850 as adequate collection rates can be obtained with the use of much smaller quantities of salt solution.

The air is supplied at a line pressure of about 40 psi. and is reduced to 25 psi. by a diaphragm type reducing valve. At 25 psi. the air passes through a water scrubber to remove oil and dirt. The pressure is then adjusted to 10 psi. by a Nullmatic Pressure Regulator.[9] (This regulator was selected as one which, under these conditions, will maintain the air pressure within 1 mm. of mercury.) The air is next saturated with water in the saturator tower and delivered to the nozzle.

TABLE VII.—PART OF DAILY LOG OF SALT FOG CABINET.

Date, February	Salt Solution As Prepared		Fog Collected	
	pH	NaCl, per cent	pH	NaCl, per cent
1	6.9	19.6	6.9	20.2
4			7.1	19.8
5			6.9	21.6
6			6.9	19.9
7			6.9	19.9
8			6.9	20.6
11	6.9	20.4	6.9	19.8
12			6.8	19.8
13			6.7	19.6
14			6.9	19.5
15			7.0	19.2
18	7.1	20.4	7.1	19.6
19			7.1	18.8
20			7.0	19.3
21			7.0	18.9
22	7.0	20.4	6.9	20.4
25			7.1	19.5
26			6.9	19.4
27			6.9	19.8
28			7.0	20.5

The temperature in this chamber is maintained between 95 F. and 96.5 F. by the heating arrangement described above. Table VII shows a portion of the daily log of this salt fog cabinet for the month of February, 1946.

By changing the direction in which the nozzle is spraying and thus the distance betweeen it and the wall which acts as a baffle, it has been possible to vary the collection rate from 0.2 to 5.6 ml. per hours.

---

[9] Moore Products Co., H and Lycoming Streets, Philadelphia, Pa.

EFFECT OF TEMPERATURE AND SPECIMEN POSITION

Many operators of salt spray fog testing equipment have asked how the results obtained with the present Method B 117 - 44T operated at 95 F. compares with similar equipment operated prior to 1944 at room temperature. To provide some information on this subject, painted panels were exposed to salt fog with a collection rate of 0.5 to 1.0 ml. per hr. Panels were exposed vertically and at 15 deg. off the vertical with the temperature controlled at 95 F. and at 72 F. (room temperature). All panels were scratched through the paint to the base metal before exposure to the salt fog. The paint creepage was determined by removing any paint which could be blown off by compressed air at 30 psi. and measuring the greatest distance from the scratch to paint film which remained intact.

TABLE VIII.—CORROSIVENESS OF THE SALT FOGS IN WHICH PAINTED PANELS WERE EXPOSED.

Temperature, deg. Fahr.	Collection Rate, ml. per hr.	NaCl, per cent	pH	Weight Lost, mg. per sq. cm.			
				Panel 1	Panel 2	Panel 3	Average
IN 25 CU. FT. CABINET AT 95 F.							
96	.6	19.1	7.5	1.86	1.94	1.91	1.90
96	.6	19.4	7.1	1.89	1.94	1.92	1.92
96	0.9	18.5	7.5	2.11	2.11	2.12	2.11
95	0.2	[a]	[a]	1.89	1.87	1.93	1.90
95	0.6	21.4	7.1	1.93	1.99	1.97	1.96
IN ROOM AT 95 F.							
95	0.1	[a]	[a]	2.07	2.32	2.44	2.26
95	0.3	25.1	6.3	1.51	1.49	1.62	1.54
95	0.8	26.3	6.3	1.60	1.56	1.68	1.61
95	0.8	28.8	6.7	1.72	1.68	1.56	1.65
95	1.1	20.4	7.1	1.94	1.88	1.87	1.90
95	0.8	20.8	7.1	1.74	1.81	1.86	1.80
IN 25 CU. FT. CABINET AT 72 F.							
72	0.1	[a]	[a]	1.04	1.14	1.19	1.12
71	0.3	18.5	7.1	1.26	1.25	1.34	1.28
68	0.2	19.4	[a]	1.38	1.24	1.32	1.31
69	0.4	18.9	7.1	1.57	1.65	1.70	1.64
68	0.5	17.3	7.1	1.57	1.57	1.35	1.50
70	0.5	21.0	7.1	1.39	1.45	1.43	1.42

[a] Collection was too small to provide adequate sample for analysis.

During the period when these panels were being tested, the corrosiveness of the salt fog was checked twice a week by means of bare steel panels exposed in groups of three.

TABLE IX.—AVERAGE MAXIMUM PAINT CREEPAGE IN SALT FOGS IN 500 HR.

(Creepage is shown in 16th of an inch. Failure denotes a creepage of one inch.)

	Vertical	15 deg. off Vertical
I Two-Coat Refrigerator Finish		
In 25 cu. ft. cabinet at 95 F.		
Phosphated steel	7	10.3
Degreaser cleaned steel	Failure at 192 hr.	Failure at 192 hr.
In salt spray room at 95 F.		
Phosphated steel	1.8	2.3
Degreaser cleaned steel	Failure at 400 hr.	Failure at 184 hr.
In 25 cu. ft. cabinet at 72 F.		
Phosphated steel	0	1.8
Degreaser cleaned steel	Failure at 500 hr.	Failure at 400 hr.
II Two-Coat Black Enamel Finish		
In 25 cu. ft. cabinet at 95 F.		
Phosphated steel	7.3	14.0
Degreaser cleaned steel	Failure at 96 hr.	Failure at 46 hr.
In salt spray room at 95 F.		
Phosphated steel	3.5	7.3
Degreaser cleaned steel	Failure at 184 hr.	Failure at 92 hr.
In 25 cu. ft. cabinet at 72 F.		
Phosphated steel	1	2.5
Degreaser cleaned steel	Failure at 264 hr.	Failure at 184 hr.

Three groups of painted panels were tested, one at 95 F. in the salt spray room, one at 95 F. in the 25-cu. ft. cabinet, and the third at 72 F. in the 25-cu. ft. cabinet. Half of each group were exposed at 15 deg. off the vertical in Micarta[10] racks and the remaining panels were exposed vertically by suspending them on strings.

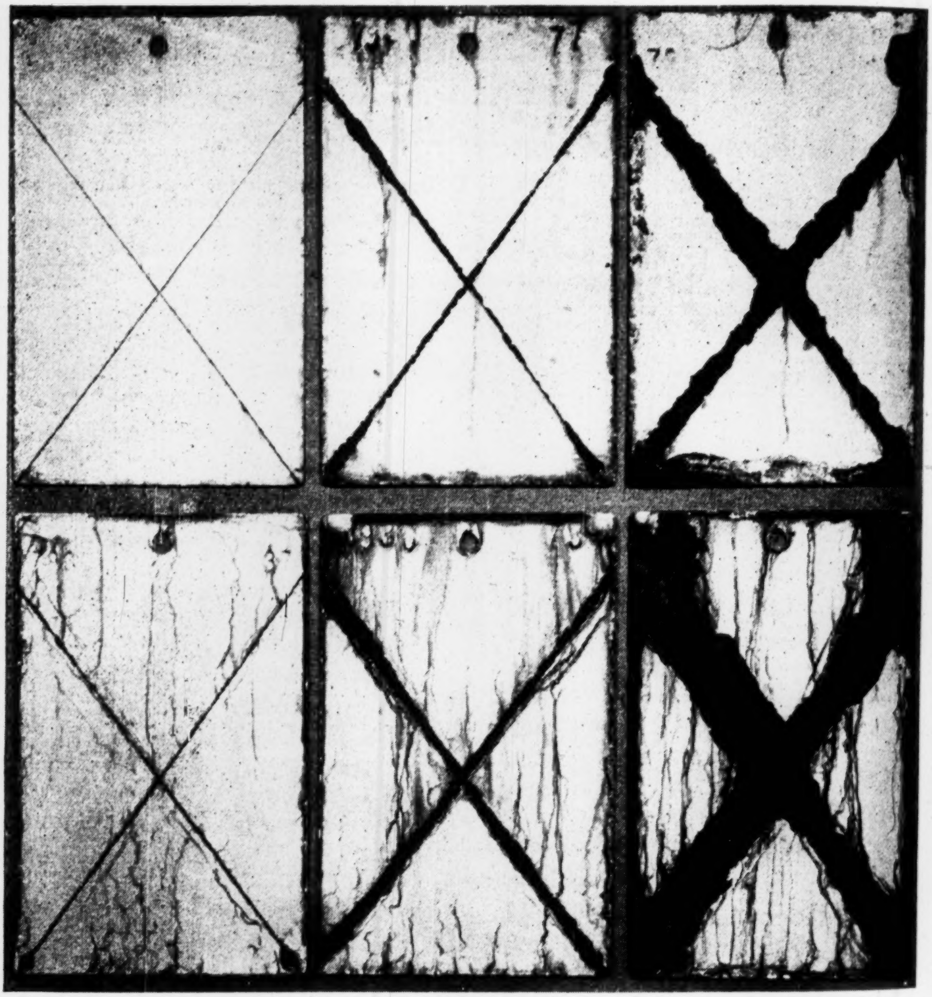
Two paint systems were used, one a two-coat refrigerator finishing system and the other a two-coat black enamel system. Half of the panels were phosphated and half were degreaser cleaned.

---

[10] Westinghouse Electric and Manufacturing Co.

The corrosiveness of the salt fogs used in these tests, as measured by the weight lost by steel panels, is shown in Table VIII. It should be noted that the salt fog in the salt spray room contained more than 23.5 per cent salt in three of the determinations and that the corrosiveness under these conditions was low. The average corrosiveness was lowest in the cabinet at 72 F. and the highest in the cabinet at 95 F.

A summary of the amount of creepage found on the panels is shown in Table IX. From these results it appears that the creepage varied with but not in proportion to the corrosiveness as measured by steel panels. The least creepage

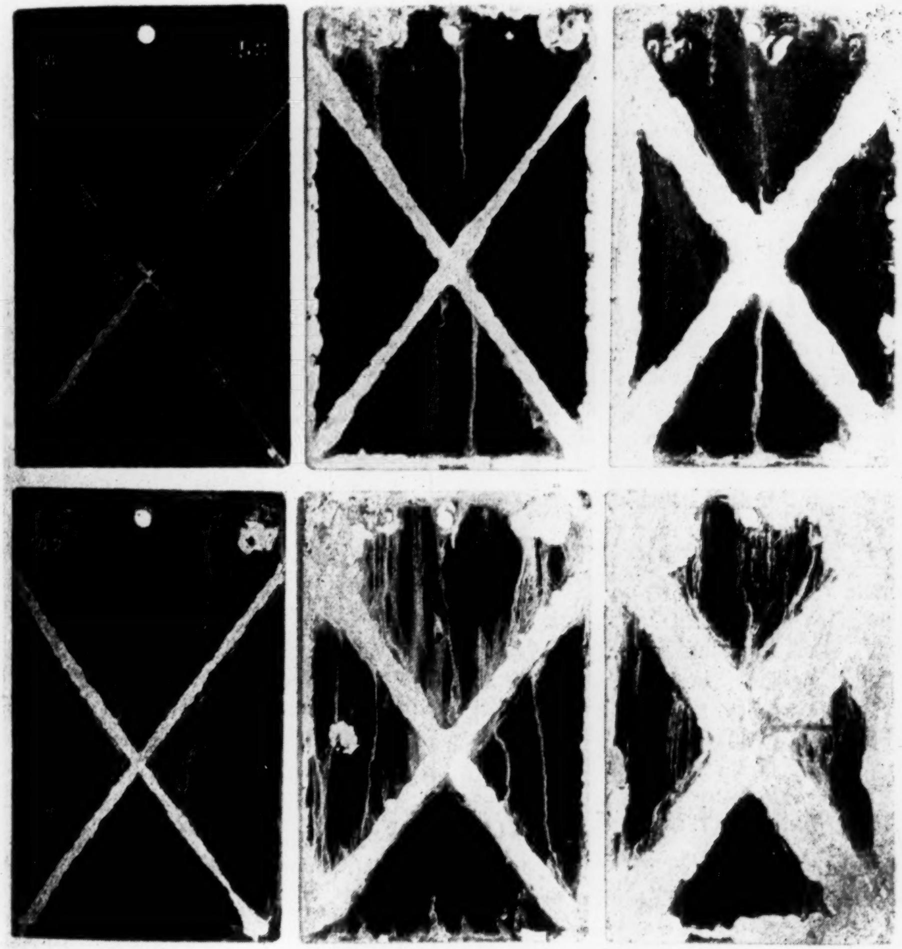


(*a*) at 72 F. in cabinet. (*b*) at 95 F. in room. (*c*) at 95 F. in cabinet.

FIG. 6.—Effect of Temperature and Specimen Position on Paint Creepage.

Top row suspended vertically. Bottom row supported at 15 deg. off vertical. Phosphated steel panels finished with a two coat refrigerator finishing system after 500 hr. exposure to salt fog test.

was found in the cabinet at 72 F. and the greatest creepage was found in the cabinet at 95 F. It is evident that the method formerly used in this laboratory (previous to 1942) of exposing panels to the salt fog vertically at room temperature produced far less creepage than does the present method of exposing the panels at 15 deg. off the vertical at 95 F.



(*a*) at 72 F. in cabinet. (*b*) at 95 F. in room. (*c*) at 95 F. in cabinet.

FIG. 7.—Effect of Temperature and Specimen Position on Paint Creepage.

Top row suspended vertically. Bottom row supported at 15 deg. off vertical. Phosphated steel panels finished with a two coat black enamel system after 500 hr. exposure to salt fog test.

The phosphated panels are shown in Figs. 6 and 7 as they appeared after 500 hr. in the salt fogs.

SUMMARY

The pH of a salt solution saturated with air rises as the temperature increases due to the lowering of the solubility of carbon dioxide in the solution. To maintain constant conditions in a salt fog chamber at 95 F., the pH should be adjusted to produce a fog which has a pH between 6.5 and 7.2. This can be accomplished by adjusting the pH of the salt solution to 6.5 to 7.2 while it is maintained at 95 F. or by adjusting the pH at room temperature until a sample has a pH of 6.5 to 7.2 after being gently boiled for 30 sec. In salt fog chambers where the salt solution is contained within the fog chamber at 95 F., no difficulty should be encountered if the solution is allowed to reach equilibrium before adjusting the pH.

Monel metal makes a satisfactory container for a neutral salt solution, but is unsatisfactory when the pH is 5.0 to 5.5 as is the case when the salt solution is kept at room temperature and adjusted so that a sample has a pH of 6.5 to 7.2 after being boiled gently for 30 sec.

There are certain critical pressures at which abnormal increases in the corrosiveness of the salt fog have been noted when the air pressure fluctuates. These critical pressures appear to be related to the size of the fluid orifice and independent of the size of the air orifice. Such increases in the corrosiveness of the fog can be avoided by controlling the pressure to avoid fluctuations greater than ±0.1 psi. or by using pressures above or below the critical pressure providing the critical pressure of the nozzle being used is known.

The salt fog chamber described provides nearly ideal heating without the necessity of providing a constant temperature room. It is possible to vary the collection rate from 0.2 ml. to 5.6 ml. per hr. without affecting the pH or concentration of the salt fog collected. The air pressure is maintained within 1 mm. of mercury.

It is essential to expose specimens to the salt fog in similar positions if results are to be comparable.

The use of a salt fog containing more than 22 per cent salt results in less paint creepage than when a fog containing 18 to 22 per cent salt is used.

The increase in the severity of the salt fog test as used in this laboratory in 1946 over the method used in 1941 can be seen by comparing the creepage on the panels suspended vertically at 72 F. and that on the panels supported at 15 deg. off the vertical at 95 F.

The use of the weight loss per square centimeter by a mild steel panel as a measure of the corrosiveness of a salt fog appears to be supported by the correlation between the weight losses in Table VIII and the paint creepage in Table IX.

REFERENCES

(1) V. M. Darsey, "The Salt Spray Test," ASTM BULLETIN No. 128, May, 1944, p. 31.

(2) S. G. Clarke, "Use of Inhibitors in Selective Removal of Metallic Coatings and Rust," *Transactions*, Electrochemical Soc., Vol. 69 pp. 131–142.

(3) "The ABC of pH Control," Sixth Edition, p. 25. LaMotte Chemical Products Co., Baltimore, Md

REPORT OF SUBCOMMITTEE VIII ON GALVANIC AND ELECTROLYTIC CORROSION

STAINLESS STEELS[1] COUPLED WITH OTHER METALS—5 YR. EXPOSURE

This report presents the data obtained in a series of tests, started in 1941, to determine the corrosion behavior of various other metals and alloys when coupled with stainless steels[1] and exposed to typical atmospheres. The data presented are for the first set of specimens which were exposed for a period of five years. A second set of specimens, now on exposure, will be continued so as to cover a period of approximately ten years, at which time this series of tests will be complete.

TABLE I.—TEST LOCATIONS.

Location	Atmosphere Classification	Date of Exposure	Exposure Period, yr.
New York, N. Y....	Industrial-Seacoast	June 15, 1941	5
Altoona, Pa.........	Industrial	Dec. 5, 1941	4.75
State College, Pa....	Rural	Dec. 4, 1941	4.75
Kure Beach, N. C...	Seacoast	June 5, 1941	5

Test Locations:

The locations and classification of the test sites are given in Table I. Four types of atmosphere are represented—namely, industrial-seacoast, industrial, rural, and seacoast. Complete information regarding the test sites, except Kure Beach, is given in the 1939 report of

TABLE II.—METALS AND ALLOYS.

Metal or Alloy	Letter	Supplied by
Stainless steel,[1] type 304...	S	The American Rolling Mill Co.
Stainless steel,[1] type 316...	U	Republic Steel Corp., Inc.
Aluminum 2S-½H.........	A	The Aluminum Company of America
Aluminum alloy 24S-T.....	E	The Aluminum Company of America
Aluminum alloy 53S-T.....	K	The Aluminum Company of America
Copper, electrolytic.......	C	Chase Brass and Copper Co.
Architectural bronze......	B	The American Brass Co.
Lead, 6 per cent antimony.	P	National Lead Co.
Zinc, Prime Western.......	Z	New Jersey Zinc Co.
Monel metal...............	M	International Nickel Co., Inc.
Iron, mild steel...........	T	Carnegie-Illinois Steel Corp.

---

[1] The word "stainless" is employed in this report as applying to austenitic chromium-nickel alloy steels. The term used in A.S.T.M. specifications for this material is "corrosion-resistant steel."

TABLE III.—CHEMICAL ANALYSIS OF METAL AND ALLOYS USED IN ATMOSPHERIC EXPOSURE TESTS ON STAINLESS STEELS COUPLED WITH OTHER METALS.

(Including data on hardness and finish).

Stainless Steels

	Type 304	Type 316
Chromium, per cent	18.89	17.95
Nickel, per cent	9.46	11.82
Carbon, per cent	0.07	0.06
Manganese, per cent	0.41	1.47
Phosphorus, per cent	0.015	0.015
Sulfur, per cent	0.015	0.019
Silicon, per cent	0.41	0.49
Molybdenum, per cent		2.90
Hardness, Rockwell	B-81	B-82
Finish, both sides	No 4	No 4

Aluminum

	2S ½H	24 S-T	53 S-T
Silicon, per cent	0.08	0.16	0.64
Iron, per cent	0.65	0.21	0.15
Copper, per cent	0.17	4.30	0.06
Manganese, per cent	0.01	0.63	0.00
Magnesium, per cent	....	1.55	1.24
Chromium, per cent	....		0.25
Finish	Commerical		

Lead, 6 per cent Antimony

Antimony, per cent	6.00
Copper, per cent	less than 0.06
Impurities, per cent	less than 0.10
Finish	Commercial

Zinc, Prime Western

Lead, per cent	0.30
Cadmium, per cent	0.15
Iron, per cent	0.01
Finish	Commercial

Monel Metal

Copper, per cent	29.02
Nickel, per cent	68.12
Carbon, per cent	0.08
Manganese, per cent	1.07
Iron, per cent	1.61
Sulfur, per cent	0.005
Silicon, per cent	0.04
Finish	Cold Rolled

Copper, Electrolytic

Antimony,percent	0.002
Iron, per cent	0.002
Lead, per cent	0.001
Nickel, per cent	0.001
Silver, per cent	0.001
Bismuth, per cent	less than 0.003
Oxygen, per cent	0.03 to 0.04
Finish	Cold rolled

Architectural Bronze

Copper, per cent	56.22
Zinc, per cent	41.36
Lead, per cent	2.30
Tin, per cent	0.11
Iron, per cent	0.01
Hardness, Rockwell	B56-60
Finish	Light annealed

Mild Steel

Carbon, per cent	0.07
Copper, per cent	0.018
Manganese, per cent	0.40
Phosphorus, per cent	0.009
Sulfur, per cent	0.032
Silicon, per cent	0.006
Iron, per cent	Bal.
Finish	Machined

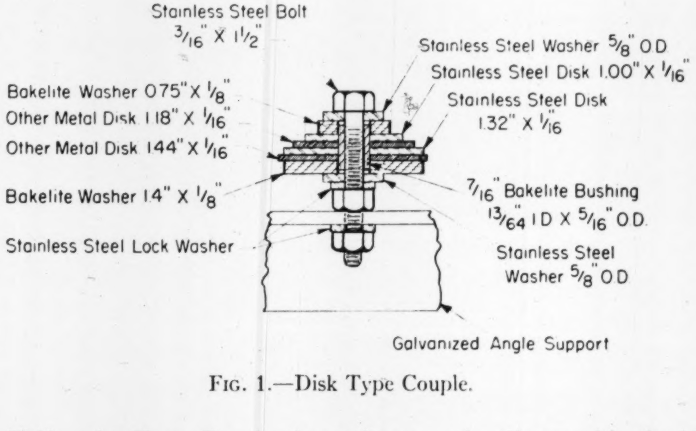

Stainless Steel Bolt $3/16'' \times 1\frac{1}{2}''$
Stainless Steel Washer $5/8''$ O D
Stainless Steel Disk 1.00" X $1/16''$
Stainless Steel Disk 1.32" X $1/16''$
Bakelite Washer 0.75" X $1/8''$
Other Metal Disk 1.18" X $1/16''$
Other Metal Disk 1.44" X $1/16''$
Bakelite Washer 1.4" X $1/8''$
$7/16''$ Bakelite Bushing $13/64''$ I D X $5/16''$ O D
Stainless Steel Lock Washer
Stainless Steel Washer $5/8''$ O D
Galvanized Angle Support

FIG. 1.—Disk Type Couple.

Committee B-3[2]. At Kure Beach the test racks are at ground level in an open coastal area in North Carolina, about 800 ft. from the Atlantic Ocean and about 20 ft. above sea level. The climatic conditions are also discussed in the 1932 report of Committee B-3.[3] These test sites are used by other A.S.T.M. Committees for atmospheric exposure tests.

---

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 247 (1939).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 214, (1932).

Metals and Alloys:

The tests include nine different metals and alloys in couple combinaton with the two types of stainless steel—namely, type 304, 18 per cent chromium-8 per cent nickel, and type 316, 18 per cent chromium-8 per cent nickel with molybdenum. In the selection of the stainless steels for these tests, Subcommittee VIII cooperated with A.S.T.M. Committee A-10 on Iron Chromium, Iron-Chromium-Nickel and Related Alloys. A list of the metals and alloys, together with the suppliers, is given in Table II. Chemical analyses, supplied by the producers of the metals are given in Table III. With the exceptions of the stainless steels and the mild steel, the tests specimens were fabricated by the producers of the metals. The stainless steel and mild steel specimens were fabricated by the Union Carbon and Carbide Laboratories.

In addition to the metals listed in the table, specimens of type 430 stainless steel coupled with 24S-T aluminum were placed on test at each of the locations in 1943. These specimens are still on exposure and are not included in this report.

It was found that the mild steel (no copper) specimens were severely corroded after one year's exposure. These specimens were removed from the test racks and mild steel with 0.05 to 0.08 per cent of copper were substituted. Consequently, the data given in this report for mild steel are for a period of one year. The data on the copper-bearing specimens are not available at this time.

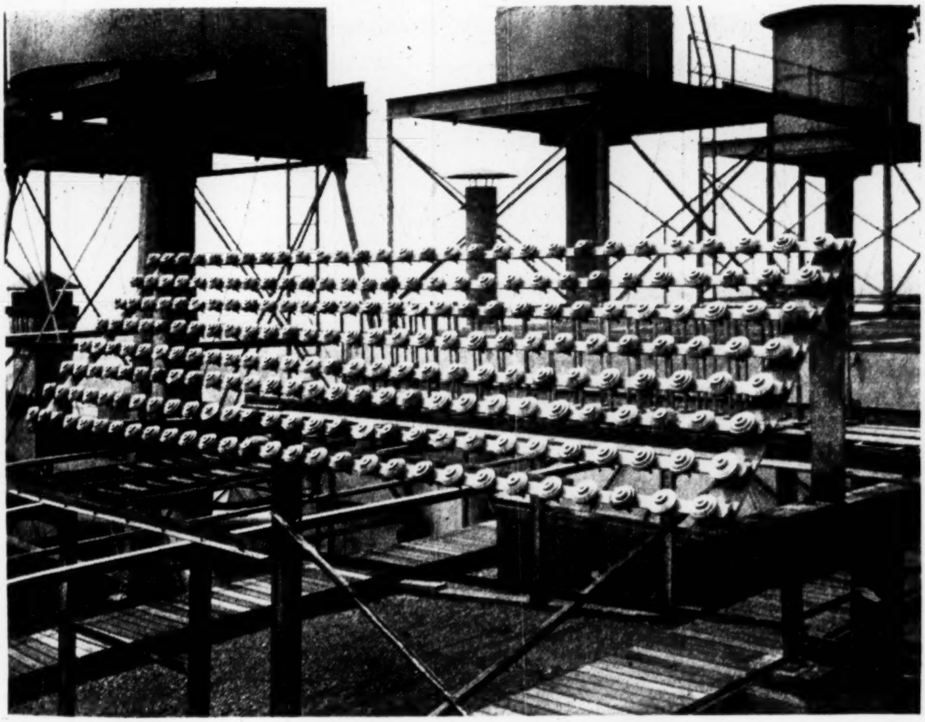


FIG. 2.—Test Racks Exposed at New York City.

Test Specimens and Couples:

The test specmens were fabricated in the form of disks of different diameters and assembled into the couple formation shown in Fig. 1. The two center disks are the test specimens of the couple. The top and bottom disks, except for inspection purposes, are not treated as test specimens. They are used primarily to produce the same type of action on both sides of the weighed specimens. The diameters of the disks were chosen so that the exposed surface area plus the edge area was the same for each weighed disk and equal to approximately 0.535 sq. in.

In addition to the couple combination illustrated, couples in which all of the disks were of the same kind of metal were tested as control couples in order to determine the normal corrosion of metal when assembled in the form of a test couple. The test specimens were weighed, initially, and the couples assembled in the laboratories of the Consolidated Edison Company of New York, Inc., after which they were distributed to the test sites.

Figures 2 and 3 show the test racks at New York and Kure Beach, respectively, and illustrate the manner in which the couples were mounted on the racks at an angle of approximately 45 deg. Two sets of couples were exposed at each location. Each set consisted of four couples of each kind; that is, four couple combinations of each metal with each type of stainless steel and four control couples for each metal. The couples in each set were placed on angle iron bars in a random pattern so as to facilitate statistical studies of the results to be obtained in the two exposure periods.

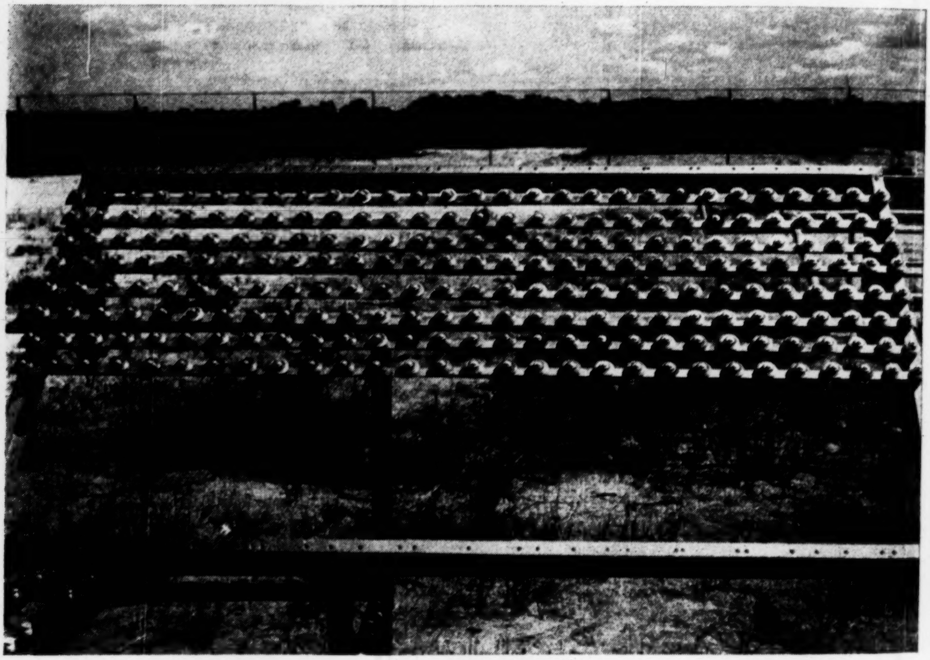


FIG. 3.—Test Racks Exposed at Kure Beach, N. C.

At approximately yearly intervals during the test period, the couples were inspected and the contact resistances checked. The ranges of the contact resistances measured on the four couples of each kind when removed at the end of the 5-yr. period are given in Table IV. With the exception of the mild steel couples which in most cases were open-circuited, and a few other cases, the contact resistance was believed to be sufficiently low to preserve the couple action.

TABLE IV.—RANGE OF COUPLE CONTACT RESISTANCE IN OHMS AT END OF 5-YR. EXPOSURE.[a]

	New York, N. Y.	Altoona, Pa.	State College, Pa.	Kure Beach, N. C.
Stainless Steel—Stainless Steel, Type 304	0.00–0.00	0.01–0.02	0.00–0.00	0.01–0.02
Stainless Steel—Stainless Steel, Type 316	0.00–0.00	0.01–0.08	0.00–0.00	0.01–0.15
Aluminum—Aluminum, 2S	0.01–0.03	0.11–9.0	0.00–0.00	0.01–1.0
Type 304—Aluminum, 2S	0.01–0.02	1.7 –9.0	0.00–0.00	0.01–0.35
Type 316—Aluminum, 2S	0.01–0.07	0.08–9.0	0.00–0.00	
Aluminum—Aluminum 24S-T	0.01–9.0*	0.15–9.0	0.00–0.00	0.01–9.0
Type 304—Aluminum 24S-T	0.01–9.0*	0.03–9.0	0.00–0.55	9.0 –9.0
Type 316—Aluminum 24S-T	0.01–5.0	0.17–9.0	0.00–2.6	9.0 –9.0*
Aluminum—Aluminum 53S-T	0.00–0.04	0.05–0.17	0.01–0.35	0.04–9.0*
Type 304—Aluminum 53S-T	0.00–0.01	0.03–0.09	0.01–0.08	0.03–3.5
Type 316—Aluminum 53S-T	0.01–0.15	2.3 –9.0	0.01–0.03	0.65–9.0*
Copper—Copper	0.00–0.00	0.00–0.00	0.00–0.00	0.00–0.00
Type 309—Copper	0.01–0.28	0.14–3.5	0.01–0.07	0.02–1.4
Type 316—Copper	0.00–0.03	0.03–0.30	0.01–0.03	0.02–3.5
Bronze—Bronze	0.00–0.00	0.05–1.4	0.00–0.00	0.00–0.00
Type 304—Bronze	0.01–0.02	0.20–0.22	0.01–0.01	0.01–0.05
Type 316—Bronze	0.01–0.08	0.04–1.9	0.00–0.01	0.02–0.02
Lead—Lead	0.00–0.00	0.00–0.00	0.00–0.00	0.00–0.00
Type 304—Lead	0.03–0.90	0.14–3.5	0.01–0.08	0.03–0.65
Type 316—Lead	0.01–0.28	0.03–0.30	0.01–9.0*	0.03–5.0*
Zinc—Zinc	0.00–0.00	0.00–0.01	0.00–0.00	0.00–0.00
Type 304—Zinc	0.00–0.01	0.17–5.0	0.01–0.03	0.01–0.02
Type 316—Zinc	0.00–0.01	0.09–9.0	0.00–0.01	0.01–0.55
Monel—Monel	0.00–0.00	0.01–0.2	0.00–0.00	0.00–0.03
Type 304—Monel	0.00–0.00	0.01–0.3	0.00–0.00	0.01–0.02
Type 316—Monel	0.00–0.00	0.01–9.0*	0.00–0.00	0.00–1.2*
Mild steel—Mild steel Type 304—Mild steel Type 316—Mild steel	Couple contacts open at end of 1-yr. exposure			

[a] 0.00 = Contact resistance less than 0.01 ohm.

* Indicates couple loose when removed from test rack.

The corrosion behavior of the various other metals coupled with stainless steel was evaluated by comparing the weight losses for the coupled specimens with the normal corrosion of the specimens in the control couples. The weight data were supplemented by visual inspections of the corroded specimens.

Cleaning and Weighing:

After a visual inspection, the specimen disks in three couples of each kind were cleaned and weighed, to the nearest milligram, to determine their weight losses. The specimens in the fourth couple were not cleaned, and are being preserved with the products of corrosion intact. Colored 35-mm. slides have been made of the specimens before and after cleaning. These photographs will be exhibited as part of the presentation of this report. Figures 4, 5, and 6 show cleaned disks with various degrees of corrosion attack.

The following procedures were followed in cleaning the disks:

Stainless Steel—The stainless steel disks were immersed in a 30 per cent (volume) solution of nitric acid at 120 to 130 F. for 5 min., removed, rinsed in hot water, scrubbed with powdered pumice and a plater's scrub brush, and rinsed. The operation was repeated, after which

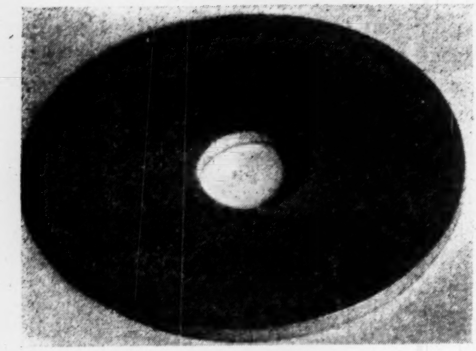


FIG. 4.—Disk Showing No Attack.

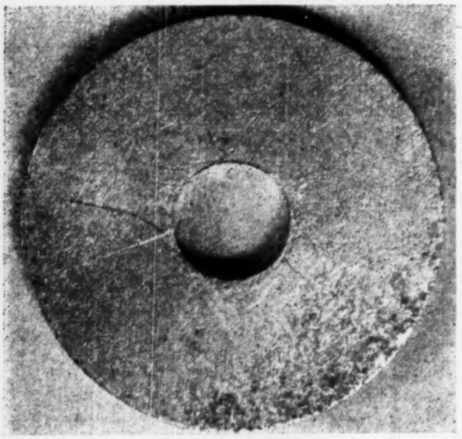


FIG. 5.—Disk Showing Medium Attack with Spotty Pitting.

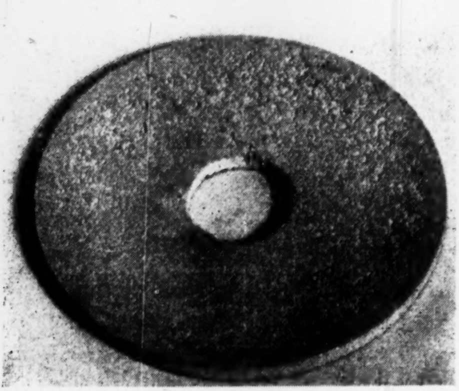


FIG. 6.—Disk Showing Heavy Attack with General Pitting.

the disks were rinsed in cold water, and in hot water, and dried.

Aluminum.— The aluminum disks were immersed in a 30 per cent (volume) solution of nitric acid at 70 F. for 10 min., removed, rinsed in hot water, scrubbed with powdered pumice and a plater's scrub brush, and rinsed. The operation was repeated, after which the disks were rinsed in cold water, rinsed in hot water and dried.

Copper.— The copper disks were cleaned for a maximum of 10 min. in a cold 10 per cent sulfuric acid solution, rinsed in cold water, scrubbed with a bristle brush, rinsed again in cold water and dried with a washed towel.

Bronze.— The bronze disks were cleaned, using the same procedure as for copper.

Lead.—The lead disks were cleaned by first treating with a solvent mixture consisting of fifty parts by volume benzol, twenty-five parts by volume acetone, twenty-five parts denatured alcohol and ten parts ethyl ether to loosen or remove organic material. They were then placed in a boiling acid ammonium acetate solution for three to five minutes and scrubbed with a stiff brush under tap water. Drying was accomplished by means of the alcohol ether system.

Zinc.—The zinc disks were immersed for 10 min. in a 10 per cent ammonium chloride solution at 175 F., rinsed and scrubbed lightly with a fiber bristle brush; they were then immersed for three minutes in sulphate-free solution of 200 g. CrO_3 in one liter of water, rinsed, again scrubbed lightly and dried.

Monel.—The monel disks were scrubbed with a mildly abrasive pumice stone, rinsed in cold water and dried.

Mild Steel.—The steel disks were immersed in a boiling solution of 20 per cent sodium hydroxide to which had been added 200 to 300 g. of zinc dust per liter. The disks were placed so that they were

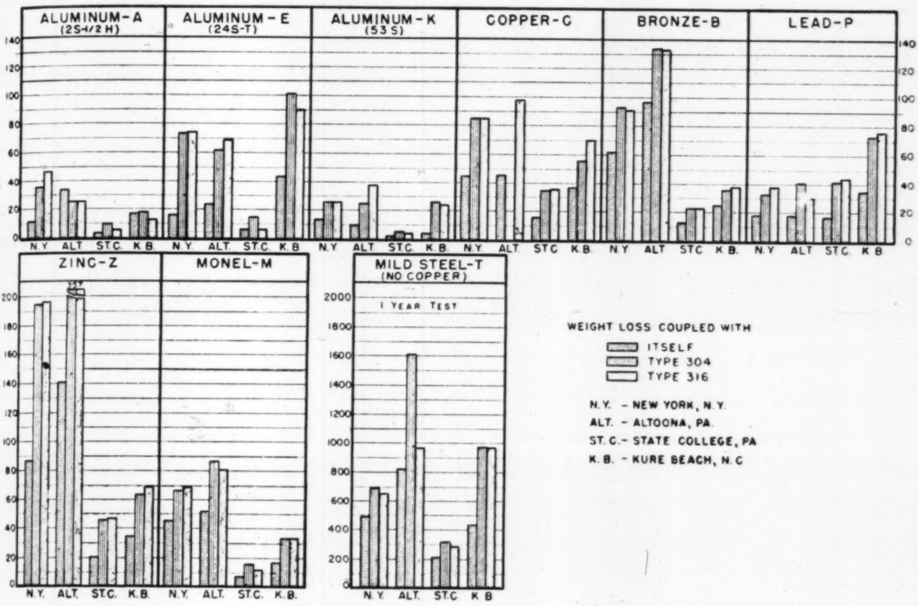


FIG. 7.—Atmospheric Corrosion of Galvanic Couples. Stainless steel coupled with other metals. Five years exposure.

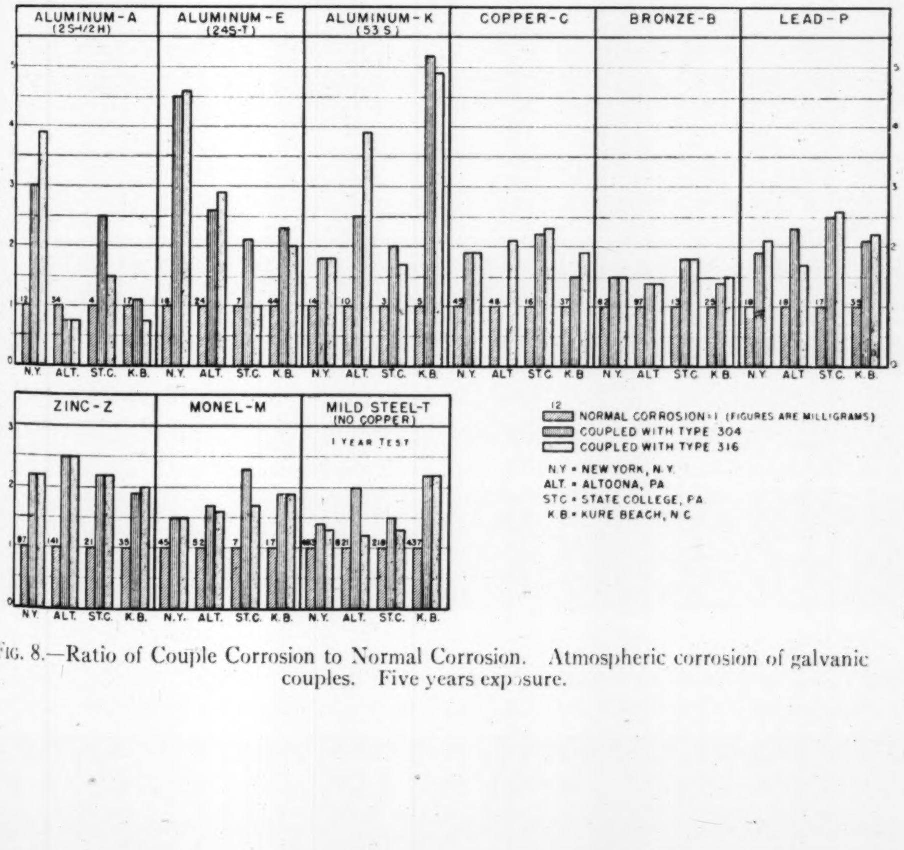


FIG. 8.—Ratio of Couple Corrosion to Normal Corrosion. Atmospheric corrosion of galvanic couples. Five years exposure.

TABLE V.—ATMOSPHERIC CORROSION OF VARIOUS METALS COUPLED WITH STAINLESS STEELS, 5 YR. EXPOSURE.

Metals and Test Locations	Average Initial Weight of Disk, g.	Average Weight Loss, mg.			Ratio of Coupled to Uncoupled Weight Loss		Visual Inspection of Corrosion			
							Exposed Edge and Surface[a]		Contact Surfaces[a]	
		Coupled with Itself	Coupled with Type 304	Coupled with Type 316	Type 304	Type 316	With Type 304	With Type 316	With Type 304	With Type 316
Aluminum (2S-1/2 H) Symbol A										
New York	2.832	12	36	47	3.0	3.9	MP —Ge—10	MP —Ge—10	MP —Ed— 4	MP —Sp— 6
Altoona	2.835	34	25	25	0.75	0.75	MP —Ge—10	MP —Ge—10	MP —Ed— 5	MP —Sp— 7
State College	2.833	4	10	6	2.5	1.5	LP —Sp— 5	LP —Sp— 5	LP —Sp— 1	LP —Sp— 2
Kure Beach	2.833	17	18	13	1.1	0.75	MP —Ge—10	MP —Ge—10	MP —Ed— 5	MP —Sp— 6
Aluminum (24 ST) Symbol E										
New York	2.946	16	73	74	4.5	4.6	MP —Ge—10	MP —Ge—10	MP —Ed— 5	MP —Sp— 7
Altoona	2.909	24	62	69	2.6	2.9	MP —Ge—10	MP —Ge—10	MP —Ed— 3	MP —Ed— 6
State College	2.936	7	15	7	2.1	1.0	LP —Sp— 5	LP —Sp— 5	LP —Sp— 1	LP —Sp— 1
Kure Beach	2.929	44	101	90	2.3	2.0	MP —Ge—10	MP —Ge—10	MP —Sp— 8	MP —Sp— 7
Aluminum (53 ST), Symbol K										
New York	2.838	14	26	26	1.8	1.8	MP —Ge—10	MP —Ge—10	MP —Ed— 2	MP —Ed— 2
Altoona	2.834	10	25	38	2.5	3.8	MP —Ge—10	MP —Ge—10	MP —Ed— 3	MP —Ed— 4
State College	2.872	3	6	5	2.0	1.7	LP —Sp— 5	MP —Sp— 5	LA —Sp— 2	LP —Sp— 1
Kure Beach	2.835	5	26	24	5.2	4.8	MP —Ge—10	MP —Ge—10	MA —Sp— 5	MA —Ed— 3
Copper, Symbol C										
New York	9.116	45	85	85	1.9	1.9	MA —Ge—10	MA —Ge—10	LP —Ed— 4	MA —Ed— 2
Altoona	9.081	46	...	98	...	2.1				
State College	9.105	16	35	36	2.2	2.3	MA —Ge—10	MA —Ge—10	LP —Ed— 2	LA —Ed— 2
Kure Beach	9.087	37	56	70	1.5	1.9	MA —Ge—10	MA —Ge—10	LP —Ed— 2	LP —Ed— 2
Bronze, Symbol B										
New York	8.746	62	93	91	1.5	1.5	MA —Ge—10	MA —Ge—10	MA —Sp— 2	MA —Sp— 3
Altoona	8.682	97	134	133	1.4	1.4	MA —Ge—10	MA —Ge—10	MA —Sp— 2	MA —Sp— 3
State College	8.715	13	23	23	1.8	1.8	LA —Ge—10	MA —Ge—10	MA —Sp— 2	MA —Sp— 1
Kure Beach	8.724	25	36	38	1.4	1.5	LA —Ge—10	LA —Ge—10	LA —Ed— 4	LA —Ed— 3
Lead, Symbol P										
New York	10.315	18	33	38	1.8	2.1	LA —Ge—10	LA —Ge—10	VLP—Ed— 1	LA —Ed— 2
Altoona	10.254	18	41	30	2.3	1.7		LA —Ge—10		VLP—Ed— 1
State College	10.289	17	42	44	2.5	2.6	LA —Ge—10	LA —Ge—10	LP —Ed— 2	LP —Ed— 3
Kure Beach	10.295	35	73	76	2.1	2.2	MA —Ge—10	MA —Ge—10	LP —Sp— 3	VLP—Ed— 1
Zinc, Symbol Z										
New York	7.461	87	195	196	2.2	2.2	MA —Ge—10	MA —Ge—10	LA —Ed— 2	LA —Ed— 3
Altoona	7.417	141	357	357	2.5	2.5	HA —Ge—10	HA —Ge—10	LA —Ed— 3	LA —Ed— 2
State College	7.445	21	46	47	2.2	2.2	MA —Ge—10	MA —Ge—10	LA —Ed— 5	LA —Ed— 5
Kure Beach	7.420	35	64	69	1.8	2.0	MA —Ge—10	MA —Ge—10	LA —Ed— 5	LA —Ed— 5
Monel, Symbol M										
New York	9.263	45	67	69	1.5	1.5	MA —Ge—10	MA —Ge—10	MA —Ed— 2	MA —Ed— 2
Altoona	9.296	52	87	81	1.7	1.6	MA —Ge—10	MA —Ge—10	MA —Sp— 2	LA —Ed— 2
State College	9.274	7	16	12	2.3	1.7	VLA—Ge—10	VLA—Ge—10	VLA—Ed— 1	VLA—Ed— 1
Kure Beach	9.277	17	33	33	1.9	1.9	MA —Ge—10	MA —Ge—10	LA —Ed— 1	LA —Ed— 2
Mild Steel (No Copper), Symbol T, 1 Year exposure										
New York	7.695	493	682	657	1.4	1.3	HA —Ge—10	HA —Ge—10	LA —Ed— 6	LA —Ed— 5
Altoona	7.592	821	1611	968	2.0	1.2	HA —Ge—10	HA —Ge—10	MA —Ge—10	LA —Ed— 5
State College	7.674	218	320	287	1.5	1.3	MA —Ge—10	MA —Ge—10	LA —Ed— 6	LA —Ed— 5
Kure Beach	7.707	437	975	974	2.2	2.2	HA —Ge—10	HA —Ge—10	MA —Ge—10	MA —Ge—10

TABLE V.—*Continued*

[a] Observations were made with a 4× magnifying glass of severity of corrosion, corrosion distribution, and portion of area corroded. Refer to Figures 4, 5, and 6 for typical illustrations of various degrees of corrosion severity.

The key for interpreting the data is as follows:

Severity of Corrosion	Corrosion Distribution	Portion of Area Corroded
HA —Heavy attack	Ge—General	1— 0 to 10 per cent
MA —Medium attack	Ed—Near Edges	2—11 to 20 per cent
LA —Light attack	Sp—Spotty	3—21 to 30 per cent
VLA—Very light attack		4—31 to 40 per cent
NA —No attack		5—41 to 50 per cent
GP —General pitting		6—51 to 60 per cent
HP —Heavy pitting		7—61 to 70 per cent
MP —Medium pitting		8—71 to 80 per cent
LP —Light pitting		9—81 to 90 per cent
VLP—Very light pitting		10—91 to 100 per cent

Thus, HA—Ge—10 indicates a heavy attack which was distributed generally over the total surface

TABLE VI.—STAINLESS STEELS, COUPLED WITH OTHER METALS—AVERAGE WEIGHT LOSS IN MILLIGRAMS.

	New York, N. Y.		Altoona, Pa.		State College, Pa.		Kure Beach, N. C.	
	With Type 304	With Type 316	With Type 304	With Type 316	With Type 304	With Type 316	With Type 304	With Type 316
Controls	(1)	(1)	(0)	(1)	(2)	(2)	(6)	(6)
Aluminum (2S-1/2 H)	1	3	2	1	2	1	0	1
Aluminum (24S-T)	3	3	1	1	2	2	2	2
Aluminum (53S-T)	2	1	0	1	1	1	1	1
Copper	0	0	......	0	0	0	0	0
Bronze	0	0	0	0	0	1	0	1
Lead	0	0	0	0	0	0	0	0
Zinc	1	1	0	0	0	1	1	1
Monel	1	1	0	0	0	0	0	0
Mild steel	0	0	0	0	0	0	0	0

in contact with the zinc dust. They were rinsed in water periodically and scrubbed with a bristle brush until the corrosion products were removed. This treatment required from thirty minutes to two hours.

Test Results:

The test results for the other metals are presented in Table V and for the stainless steel specimens in Table VI. The weight losses in milligrams for the control specimens and the coupled specimens are shown by the graphs plotted in Fig. 7, and the weight changes resulting from the coupled combinations, expressed as ratios to the normal corrosion, are shown graphically in Figure 8.

The second column of Table V gives the average initial weights in grams, of the other metal specimens. The variation in the weights of the individual specimens did not exceed two per cent of the average weight. The third, fourth and fifth columns give the weight losses in milligrams for the specmens of the other metal coupled with itself and with the type 304 and 316 stainless steels. The sixth and seventh columns give the ratio of coupled to uncoupled weight losses. The column headed "Visual Inspection" gives the results of a visual inspection of the cleaned specimens expressed in terms of the inspection code given at the bottom of the table. The inspection was made with a 4× magnifying glass.

Since this is essentially a progress report and presents the data on the first of two sets of specimens, no attempt is made at this time to include a discussion of the results.

Respectfully submitted on behalf of the subcommittee,

L. J. GORMAN,
Chairman.

REPORT OF COMMITTEE B-4*

ON

ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS

Committee B-4 on Electrical Heating, Resistance and Related Alloys held three meetings since its previous report: in Atlantic City, N. J., on June 18 and 19, 1947; in Philadelphia, Pa., on November 12 and 13, 1947, and on February 11 and 12, 1948.

The committee consists of 71 members, of whom 29 are classified as producers, 35 as consumers, and 7 as general interest members.

The election of officers for the ensuing two years resulted in the selection of the following:

Chairman, S. A. Standing.
Vice-Chairman, E. I. Shobert, II.
Secretary, F. E. Bash.
Corresponding Secretary, S. Umbreit.

NEW TENTATIVE

The committee recommends that the new Tentative Method of Test for Modulus of Elasticity of Thermostat Metals (Cantilever Method)[1] be accepted for publication as tentative.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends that the Standard Method of Test for Resistivity of Metallic Materials (B 63 – 36) and the Standard Method of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 – 42) be revised, as appended hereto,[2] and reverted to tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating Materials (W. A. Gatward, chairman).—A five-year average of accelerated life tests made in accordance with the Standard Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 – 39) indicate an average life of 145 hr. for tests made in July and August but 195 hr. for tests made in December and January. These results are not conclusive, as voltage regulation, humidity, and variations in ambient temperature affect the hours of life and reproducibility of life tests, and consequently the subcommittee is continuing its investigation of the method.

Subcommittee II on Electrical Tests (P. H. Dike, chairman) prepared the revision of Method B 63 referred to earlier in this report.

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (J. W. Harsch, chairman) is working on a suggested program of cooperation with the Alloy Casting Institute in an attempt to revise the Tentative Specifications for Nickel-Chromium-Iron Alloy Castings (35 – 15 Class) for High-Temperature Service (B 207 – 46 T) to make them more satisfactory. In the meantime, these specifications and the Tentative Specifications for Chromium-Nickel-Iron Alloy Castings (25 – 12 Class) for High-Temperature Service (B 190 – 45 T) are being continued as tentative.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman) has concluded, after investigation, that the cantilever method is the most practical currently available for testing the modulus of elasticity of thermostatic metals, and accordingly based the new Tentative Method of Test for Modulus of Elasticity of Thermostat Metals on this method. However, the subcommittee is also working on the development of an apparatus for determining the modulus of thermostatic metals in tension.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] This method was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

[2] These revised methods were accepted by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman).—The various subsections of Section A on Cathode Materials have been working on the following projects: Check analyses of a number of heats of cathode nickel and preparation of a new form for reporting analyses; a revised tentative manual for melt approval of cathode nickel; summary of miscellaneous information regarding chemical analyses of nickel for silicon, manganese, magnesium, carbon, and sulfur, to permit correlation with tube test data; preparation of proposed spectrochemical methods of analysis of nickel in oxide form for copper, iron, magnesium, manganese, silicon, and titanium as well as colorimetric methods for copper, iron, and manganese; organization of a subgroup to discuss reducing agents in normal and active cathode nickel (at a meeting of which it was decided that the exact analyses of carbon, magnesium, and silicon are necessary); discussion of the extensive diode life tests of cathode material under pulse conditions; furtherance of the study of interface between the cathode and the coating; checking of a series of six lots with variable carbon content in which no significant differences that could not be attributed to variations in coating or processing were obtained; presentation of papers[4] describing the work of this section which were given at the Institute of Radio Enginners Convention in New York, N. Y., on March 24, 1948; and preparation and testing of special heats of nickel with controlled amounts of impurities.

Section B in cooperation with Committee A-6 on Magnetic Properties completed the Tentative Method of Test for Permeability of Paramagnetic Materials (A 34 - 47 T).[5]

Subcommittee IX on Methods of Tests for Alloys in Controlled Atmospheres (P. H. Brace, chairman) is working on the testing of samples of nickel oxide and chromium trioxide under various furnace atmospheres together with pieces of 80-20 furnace ribbon in another effort to trace the phenomenon of "green rot" in electric furnaces using controlled atmospheres.

Subcommittee X on Contact Materials (F. E. Carter, chairman).—Section A has actively continued its investigation of electrical characteristics of contacts. Section B cooperated with sub-committee II in the preparation of the revision of Method B 63 referred to earlier in this report. Section C has prepared an article on Standardization of Electrical Contacts which will be published in *Electrical Engineering*. Section E prepared the 1947 Supplement to Bibliography and Abstracts on Electrical Contacts.

In order to broaden the field, Section F has been formed with A. H. Lamb as chairman to study micro-current contacts. Invitations to join in this work are being issued to laboratories studying contacts but not now members of Subcommittee X.

This report has been submitted to letter ballot of the committee which consists of 71 members, 56 members returned their ballots, of whom 53 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. HARSCH,
Chairman.

F. E. BASH,
Secretary.

---

[4] Abstracts of these papers are printed in the ASTM BULLETIN, No. 152, May, 1948.

[5] 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

REPORT OF COMMITTEE B-5* ON COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year at Pittsburgh, Pa., on November 7, 1947, and at Washington, D. C., on March 5, 1948. At these sessions the Advisory Committee and Subcommittees W-1, W-2, W-3, W-4, G-1, and G-3 also met. Subcommittee F-1 met at Washington.

During the year 20 members were added to the committee and there were 13 removals. At the present time the committee consists of 137 members, of whom 117 are voting members; 51 are classified as producers, 48 as consumers, and 18 as general interest members.

Subsequent to the 1947 Annual Meeting, there was presented to the Society through the Administrative Committee on Standards the proposed Tentative Classification of Copper which was prepared by a joint committee representing Committee B-5, Committee B-1 on Wires for Electrical Conductors, and Committee B-2 on Non-Ferrous Metals and Alloys. The new tentative classification was accepted by the Standards Committee on March 5, 1948, and has been issued under the designation B 224 - 48 T.

At the meeting on March 5, 1948, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, G. H. Harnden.
First Vice-Chairman, H. C. Ashley.
Second Vice-Chairman, J. J. Kanter.
Secretary, V. P. Weaver.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting revisions in 12 tentative specifications, revisions in 7 standards for immediate adoption, tentative revisions in 5 standards, and 10 tentatives are recommended for adoption as standard. The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF THE ADVISORY COMMITTEE

The following revisions in the regulations of the committee were approved by letter ballot:

Article VI, Section 1.—Revise the second sentence of the second paragraph to read as follows: "These groups shall be: Group W—Wrought Products; Group F—Cast Products; Group G—General."

Article VII, Sec. 2.—In the first sentence of the second paragraph revise to read as follows: ". . . originating in a Group W or a Group F subcommittee."

Standard Specifications B 42, B 43, B 66, B 67, B 99, B 100, B 134 and B 140 are being recommended for approval as American Standard by the American Standards Association.

ACTIVITIES OF SUBCOMMITTEES

The following is the report of the several subcommittees during the year.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

The actions are given in detail in the Appendix.

Subcommittee W-1 on Plate, Sheet, and Strip (R. A. Frye, chairman, S. A. Rosecrans, secretary) recommended that revisions be incorporated in three tentative specifications: B 36, B 122, and B 152. Revisions for immediate adoption were recommended in Standard Specifications B 171.

Among the subjects being studied by the subcommittee are the revision of requirements for mechanical properties in Specifications B 194 and B 195, the writing of specifications to cover aluminum bronze plate and sheet for A.S.M.E. Boiler Code requirements, the revision of copper limits for alloys Nos. 4 and 6 of Specifications B 121, the addition of rolled bar to plate specifications and the narrowing of tin limits and the addition of grain size limits in Specifications B 103.

Subcommittee W-2 on Rods, Bars, and Shapes (H. C. Ashley, chairman) recommended that revisions be incorporated in seven tentative specifications: B 21, B 133, B 139, B 150, B 151, B 187, and B 196. Revisions for immediate adoption were recommended in Standard Specifications B 124 and B 138. Tentative revisions were recommended in five standard specifications: B 12, B 16, B 98, B 138, and B 140.

A task group (V. P. Weaver, chairman) prepared the revisions incorporating Methods of Test B 220 in the rod and bar specifications in place of Methods of Test E 8.

Among the subjects being studied by the subcommittee are the possible deletion of bend tests in rod and bar specifications, the inclusion of additional size range in Specifications B 133 and B 187, the revision of requirements for mechanical properties in Specifications B 150, the revision of elongation requirements in rod and bar specifications, the narrowing of tin limits in Specifications B 139, and the revision of chemical limits for alloy No. 11 in Specifications B 124.

Subcommittee W-3 on Wire and Wire Rod (B. J. Sirois, chairman) recommended revisions for immediate adoption in Standard Specifications B 134.

The subcommittee is studying the narrowing of tin limits and the addition of grain size limits in Specifications B 159.

Subcommittee W-4 on Pipe and Tube (Alan Morris, chairman, R. S. Pratt, secretary) recommended that revisions be incorporated in Tentative Specifications B 75. Revisions for immediate adoption were recommended in Standard Specifications B 88 and B 111.

A task group (J. S. Rodgers, chairman) prepared the minimum limits for inhibitors in the revisions for chemical limits in Specifications B 111. A task group (G. H. Harnden, chairman) prepared the revisions of pressure test requirements in pipe and tube specifications.

Among the subjects being studied by the subcommittee are the development of requirements for dehydrated and sealed refrigeration tubes in Specifications B 68, the revision of dimensional tolerances in Specifications B 111, and the deletion of weight tolerance in Specifications B 88. The temper requirements of Specifications B 75 are being reviewed as to their adequacy.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman, C. S. Cole, secretary) recommended revisions for immediate adoption in Standard Specifications B 52. Specifications B 132, B 145, B 146, B 148, B 149, and B 198 were recommended for adoption as standard without revision. With minor revisions Specifications B 30, B 143, B 144, and B 147, were also recommended for adoption as standard. It was recommended that Recommended Practice B 208 be continued as tentative pending revisions being formulated by the test bar task group.

A task group (William Romanoff, chairman) prepared the revisions of Specifications B 30. A task group (N. H. Murdza, chairman) prepared the revisions for Specifications B 149.

The subcommittee has under way the revision of sampling requirements in Specifications B 30 and the preparation of appendices for Specifications B 147 and B 198 to cover manufacturers' published composition limits for specific alloys. A task group continues its extensive study of test bars and test bar practices.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman, J. P. Guerard, secretary) recommended that revisions be incorporated in Tentative Method B 220 as prepared by a task group (S. A. Rosecrans, chairman). It also recommended, as prepared by a task group (J. P. Guerard, chairman), a proposed Method of Test for the Tension Testing of Thin Sheet Metal (0.002 to 0.010 in. Thickness), which will be submitted to Committee E-1 on Methods of Testing.

The subcommittee is studying the effect of speed of testing on tensile properties and the relation of Rockwell hardness to thickness of specimen.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) recommended the revision of length tolerances in Specifications B 187, and the deletion of straightness tolerances for shafting in Specifications B 21, B 138, B 139, and B 150. Revisions in the straightness tolerances in Specifications B 187 and in the tolerances in Specifications B 88 and B 111 have been proposed. The subcommittee is studying the revision of dimensional tolerances in all wrought product specifications to correspond with current CABRA standards.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) recommended the incorporation of a note as to significance of dimensional tolerances in wrought product specifications.

It is continuing a study of the correlation of the chemical limits and of the nomenclature and classification of wrought alloys under the jurisdiction of the committee. It is also studying the preparation of a recommended form for Committee B-5 standards, and is preparing a history of the committee.

This report has been submitted to letter ballot of the committee which consists of 117 voting members; 93 members returned their ballots, of whom 87 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1946 Book of A.S.T.M. Standards, Part I-B or in the 1947 Supplement to Book of A.S.T.M. Standards, Part 1-B.

Revisions of Tentative Specifications

The committee recommends that the following 12 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Naval Brass Rods, Bars, and Shapes (B 21 – 47 T):

Section 9.—Revise Paragraph (*a*) to read as follows: "In the tension test all material shall be pulled in full size when practicable. Full size or machined test specimens shall be as specified in the Tentative Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes (A.S.T.M. Designation: B 220)."

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Table VII.—Delete straightness tolerances for piston finish rods and shafting.

Section 14.—In Paragraph (*b*) delete reference to straightness tolerances.

Revise Paragraph (*c*) to read as follows: "The straightness tolerances for shafting and piston finish rod are subject to agreement between the manufacturer and the purchaser."

Tentative Specifications for Brass Plate, Sheet, and Strip (B 36 – 47 T):

Section 4.—For alloy No. 8 revise the copper limits from "64.0 to 68.0" to read "64.0 to 68.5" per cent.

Tentative Specifications for Seamless Copper Tubes (B 75 – 47 T):

Section 9.—In Paragraph (*a*) between the words "tube shall" in the second line, insert "selected for purpose of test."

In Paragraph (*c*) in the sixth line revise "60 to 100" to read "not less than 60" psi.

In Paragraph (*d*), Item (*2*) revise the third line to read "at not less than 60 psi. air pressure while immersed in water."

Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet and Strip (B 122 – 47 T):

Section 10.—Add alloy No. 4 to each nominal grain size.

Table III.—After alloy No. 3 add the following:

Alloy No. 4 (55 Cu, 18 Ni, 27 Zn)

Temper, Nominal Grain Size	Rockwell Hardness					
	B Scale		F Scale		Superficial 30 T	
	Min.	Max.	Min.	Max.	Min.	Max.
0.070-mm.	29	45	72	83	35	46
0.035-mm.	37	60	76	91	41	57
0.015-mm.	47	73	84	98	47	65

Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 – 47 T):

Section 13.—Revise Paragraph (*a*) to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 15.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 – 47 T):

Section 2.—Add a new Item "(2) Temper (Section 6; Table II)" and renumber subsequent items accordingly.

Section 8.—Revise Paragraph (*a*) to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 10.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Table VII.—Delete straightness tolerances for piston finish rods and shafting.

Section 13.—In Paragraph (*b*) delete reference to straightness tolerances.

Revise Paragraph (*c*) to read as indicated above for Section 14 (*c*) in Specifications B 21.

Tentative Specifications for Aluminum Bronze Rods, Bars, and Shapes (B 150 – 47 T):

Section 9.—Revise Paragraph (*a*) to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Table VIII.—Delete straightness tolerances for piston finish rods and shafting.

Section 14.—In Paragraph (*b*) delete reference to straightness tolerances.

Revise Paragraph (*c*) to read as indicated above for Section 14 (*c*) in Specifications B 21.

Tentative Specifications for Copper-Nickel-Zinc Alloy Rods and Bars (B 151 – 47 T):

Section 9.—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 – 47 T):

Section 1.—Revise type designations A, B, C, D1, D2, D3, and E to read respectively ETP, DHP, OF, STP, DPS, OFS and ATP; also add type FRTP (Fire Refined Tough Pitch Copper) after type A. Make corresponding revisions in Section 2, Items 1 (*a*) and 3; Sections 4, 12, 13 and 14.

Section 4.—After type A add type FRTP with chemical limits as given in Section 2 of Specifications B 216.

Tentative Specifications for Copper Bus Bars, Rods, and Shapes (B 187 – 47 T):

Section 8 (a).—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 10.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Section 11 (c).—Revise to read as follows:

> (*c*) *Specified Lengths.*—When exact lengths are ordered, the lengths shall be not less than the ordered length and shall not exceed it by more than the following amounts:

Specified Length, ft.	Tolerances, in.[a]
Up to 6, incl.	1/8
Over 6 to 15, incl.	1/4
Over 15	3/8

[a] All tolerances are plus; minus tolerances are not permissible.

Table VI.—Revise to read as shown in the accompanying Table I.

Tentative Specifications for Beryllium-Copper Alloy Rod and Bar (B 196 – 47 T):

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Tentative Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes (B 220 – 47 T):

Section 3.—Change the last sentence to read as follows: "The testing machine shall be of such capacity as to be capable of fracturing either the full size test specimen referred to in Section 5 or the standard machined test specimen shown in Fig. 2."

Section 5 (b).—Change the last sentence to read "In no case shall the material be machined to less than the diameter shown in Fig. 2."

Section 5 (c), Item (2).—Revise to read as follows:

(*2*) The width of the specimen between the gage marks may be reduced with abrasive cloth or paper, or by machining sufficiently to facilitate fracture within gage marks, but in no case shall the reduced width be less than 90 per cent of the original. The edges of the mid-length of the reduced section not less than $\frac{3}{4}$ in. in length shall be parallel to each other and to the longitudinal axis of the specimen within 0.002 in. Fillets with $\frac{3}{8}$ in. radius preferably, but not less than $\frac{1}{8}$ in. radius shall be used at the ends of the reduced sections.

TABLE I.—SCHEDULE OF SHORT LENGTHS.
(Applicable to stock lengths with short lengths included.)

Rounds, Hexagons, Octagons, and Squares	Rectangles	Indicated Lengths,[b] ft.	Shortest Permissible Length,[c] per cent of indicated length	Maximum Permissible per cent of Lot by Weight of Short Lengths
Diameter or Distance Between Parallel Surfaces, in.	Area,[a] sq. in.			
Up to ½, incl.	Up to 0.250, incl.	10, **12**, 14	75	20
Over ½ to 1, incl.	Over 0.250 to 1, incl.	10, **12**, 14	70	30
Over 1 to 1½, incl.	Over 1 to 2.25, incl.	8, **10**, 12	60	40
Over 1½ to 2, incl.	Over 2.25 to 4, incl.	8, **10**, 12	50	45
Over 2 to 3, incl.	Over 4 to 9, incl.	6, **8**, 10	40	50

[a] Width times thickness, disregarding any rounded corners or edges.
[b] For other indicated lengths, consult the manufacturer. Lengths given in boldface type are preferred and most commonly used.
[c] Expressed to nearest 0.5 ft.

Section 5 (a), Item (2).—Revise to read as follows:

(*2*) The surface of the specimen between the gage marks may be smoothed with abrasive cloth or paper, or machined sufficiently to facilitate fracture within the gage marks. For material over 0.188 in. in diameter or distance between flats, the diameter or distance between flats may be reduced by not more than 0.010 in. For material 0.188 in. and under in diameter or distance between flats, the cross-sectional area may be reduced to not less than 90 per cent of the original area. As an alternative, hexagonal or octagonal rod over 0.188 in. between flats may be turned to a round having a diameter no smaller than 0.010 in. less than the original distance between flats. Hexagonal or octagonal rod 0.188 in. and under between flats may be turned to a round leaving a cross-sectional area no smaller than 90 per cent of the area of the maximum inscribed circle. Fillets with $\frac{3}{8}$ in. radius preferably, but not less than $\frac{1}{8}$ in. radius shall be used at the ends of the reduced sections.

Section 5 (d).—Change the last sentence to read: "In no case shall the material be machined to less than the diameter shown in Fig. 2." Add a new sentence to read: "Fillets with $\frac{3}{8}$ in. radius preferably, but not less than $\frac{1}{8}$ in. radius shall be used at the ends of the reduced sections."

Section 6 (a).—In line 5, delete "0.505 in. in diameter."

Section 7.—Substitute the following for the third sentence: "Gage marks shall be stamped lightly with a punch, scribed lightly with dividers, or drawn with ink as preferred. For material $\frac{1}{8}$ in. and under in thickness, gage marks located with the aid of layout dope will minimize the possibility of fracture in the gage marks."

Delete Paragraphs (*a*) and (*b*).

Section 11.—Delete the second sentence.

Section 12.—Change the first sentence to read: "For purposes of acceptance or rejection the yield strength shall be determined by either of the methods described in Paragraphs (*a*) and (*b*).

Section 12 (*b*).—Delete the first two lines and replace by "For material whose stress. . .".

Section 14 (*b*).—Revise the first sentence to read: "If any part of the fracture takes place outside of the middle two-thirds of the gage length or in a punched or scribed mark within the reduced section, the elongation value obtained may not be representative of the material."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in 7 specifications as indicated below and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Phosphor Copper (B 52 – 46):

Section 5.—Combine Paragraphs (*a*) and Item (*1*) as "(*a*) Ingots and Slabs, Grade A,", changing the lot size from "100" to "1000" lb. and insert a new second sentence to read as follows: "If the lot is less than 1000 lb., one pound of material shall nevertheless be taken." Delete the third sentence.

Delete the present Item (*2*) and add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as (*c*):

> (*b*) *Ingots and Slabs, Grade B.*—For each 1000 lb. of the lot three slabs or ingots shall be selected as the sample. If the lot is less than 1000 lb., three slabs or ingots shall, nevertheless, be taken. Drillings shall be taken by drilling completely through the ingot or slab in at least three places; namely, at the center and near the edges. The drillings shall be mixed and quartered.

Standard Specifications for Copper Water Tube (B 88 – 47):

Section 10.—Revise Paragraphs (*c*) and (*d*) Item (*2*) to read as indicated above for Paragraphs (*c*) and (*d*) Item (*2*) of Section 9 in Specifications B 75.

Section 11 (*a*).—In the second line revise "0.5" to read "0.2" per cent.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 47):

Table I.—For admiralty metal and aluminum brass, revise the arsenic, antimony, and phosphorus maximum values of "0.10" to read "0.02 to 0.10" per cent. For 70-30 copper-nickel, add a footnote referring to iron content to read as follows: "When specified by the purchaser the iron content shall be 0.25 to 0.60 per cent." For copper, revise the maximum phosphorus content of "0.035" to read "0.04" per cent.

Section 11.—Revise to read as in Section 9 (*a*) in Specifications B 75 – 47 T omitting the phrase "except as provided in Paragraphs (*b*) to (*d*)" and substitute "7,000" for "6,000" psi. as the value for fiber stress and unit stress.

Standard Specifications for Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 47):

Table I.—For alloy No. 1 revise the maximum lead content of "0.20" to read "0.30" per cent.

Delete the requirements for "Sum of Named Elements" for alloys Nos. 1 to 6, inclusive and substitute the following: "Other Impurities, max., per cent, No. 1, 0.35; No. 2, 0.5; No. 3, 0.1; No. 4, 0.1; No. 5, 0.1; and No. 6, 0.1."

Add the following footnote to this table: "For alloys Nos. 1 to 6, inclusive, analysis shall regularly be made only for the elements specifically mentioned in this table with the exception of zinc which shall be determined by difference. If the presence of other elements is suspected or indicated in the course of routine analysis, further analysis shall be made either chemically or spectrographically to determine that the total of these other elements is not in excess of the limits specified."

Standard Specifications for Brass Wire (B 134 – 47):

Table I.—For alloy No. 7 revise the copper limits of "63.0 to 67.5" to read "63.0 to 68.5" per cent.

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 47):

Table IX.—Delete straightness tolerances for piston finish rods and shafting.

Section 14.—In Paragraph (*b*) delete reference to straightness tolerances.

Revise Paragraph (*c*) to read as indicated above for Section 14 (*c*) in Specifications B 21.

Standard Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 47):

Table II.—Revise the present subheading to read as follows: "Thickness Tolerances, plus and minus,[b] in., for Diameters or Widths," retaining the present footnote *b*.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends revisions of 5 standards for publication as tentative as indicated below.

Standard Specifications for Copper Rods for Locomotive Staybolts (B 12 – 47):

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Standard Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 47):

Section 9 (a).—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 47):

Section 8 (a).—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 10.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 47):

Section 9 (a).—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 11.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 47):

Section 7.—Revise to read as indicated above for Section 9 (*a*) in Specifications B 21.

Section 9.—Replace reference to Standard Methods E 8 by a reference to Tentative Methods B 220.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following 6 tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 – 46 T),

Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 46 T),

Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 46 T),
Aluminum Bronze Sand Castings (B 148 - 46 T),
Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 - 46 T),
Silicon-Bronze and Silicon-Brass Sand Castings (B 198 - 46 T).

The committee recommends that the following 4 tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 45 T):

Table II.—For alloy 2B revise minimum zinc from "3.5" to read "3.0" per cent.

For alloys 3A, 3D, and 3E revise the maximum nickel from "0.50" to read "0.75" per cent.

For alloys 8B and 8C revise maximum tin "0.50" to read "0.10" per cent.

Table III.—For alloy 8B, revise elongation value from "20" to read "18" per cent.

Tentative Specifications for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 46 T):

Table II.—For alloy 2B change the value for minimum zinc from "3.0" to read "2.5" per cent.

Tentative Specifications for High-Leaded Tin-Bronze Sand Castings (B 144 - 46 T):

Table II.—For alloys 3A and 3E, change the maximum nickel value from "0.50" to read "0.75" per cent.

Tentative Specifications for High-Strength Yellow Brass (Manganese Bronze) and Leaded High Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 - 46 T):

Table II.—For alloys 8B and 8C, change the maximum tin requirement from "0.5" to read "0.20" per cent.

Table III.—For alloy 8B change the elongation requirement from "20" to read "18" per cent.

EDITORIAL CHANGE IN TENTATIVES AND STANDARDS

In the indicated sections of the tentatives and standards listed below, add a note to read as follows: "Note.—The dimensional tolerances given in these specifications are absolute. Departure from the specified dimensions by any amount greater than the indicated tolerance may be cause for rejection."

	Section		Section
B 19 - 47 T....	14	B 12 - 47......	12
B 21 - 47 T....	12	B 13 - 41......	14
B 36 - 47 T....	14	B 14 - 18......	13
B 75 - 47 T....	16	B 16 - 47......	12
B 121 - 47 T....	14	B 42 - 47......	14
B 122 - 47 T....	14	B 43 - 47......	14
B 129 - 47 T....	8	B 68 - 47......	13
B 130 - 47 T....	14	B 88 - 47......	15
B 131 - 47 T....	9	B 96 - 47......	9
B 133 - 47 T....	17	B 97 - 47......	10
B 135 - 47 T....	15	B 98 - 47......	11
B 139 - 47 T....	11	B 99 - 47......	10
B 150 - 47 T....	12	B 100 - 47......	8
B 151 - 47 T....	12	B 103 - 47......	12
B 152 - 47 T....	17	B 111 - 47......	15
B 159 - 47 T....	10	B 124 - 47......	7
B 169 - 47 T....	10	B 134 - 47......	12
B 187 - 47 T....	11	B 138 - 47......	12
B 188 - 47 T....	12	B 140 - 47......	10
B 194 - 47 T....	12	B 171 - 47......	9
B 195 - 47 T....	12		
B 196 - 47 T....	12		
B 197 - 47 T....	13		
B 206 - 47 T....	13		

REPORT OF COMMITTEE B-6*
ON
DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 19, 1947, and one in Washington, D. C., on March 3, 1948, with an attendance of 38 and 41, respectively.

The members of Committee B-6 contributed $519.58 to the A.S.T.M. Building Fund as the direct result of an appeal made by this committee for such donations at the 1947 Annual Meeting.

There is a balance of $2599.55 in the funds of Committee B-6 as of December 31, 1947.

At the present time, the committee consists of 89 members, of whom 41 are classified as producers, 24 as consumers, and 24 as general interest members.

I. Revision of Tentatives

The committee recommends the following revisions in three tentative specifications:

Tentative Specifications for Aluminum-Base Alloy Die Castings (**B 85 – 46 T**):[1]

Section 1.—Change to read as follows: "These specifications cover aluminum-base alloy die castings. Ten alloy compositions are specified and are designated as shown in Table I."

Table I.—Add the following chemical requirements for the new alloy G2:

Magnesium, per cent	7.5 to 8.5
Iron, max., per cent	1.8
Manganese, max., per cent	0.3
Silicon, max., per cent	0.3
Copper, max., per cent	0.2
Tin, max., per cent	0.1
Zinc, max., per cent	0.1
Nickel, max., per cent	0.1
Other Constituents, Except Aluminum, max., (Total), per cent	0.2
Aluminum, per cent	remainder

Appendix.—Delete the last sentence and substitute the following: "In the tension testing of die-cast aluminum-alloy specimens, the rate of stressing up to the yield strength shall not exceed 100,000 psi. per min. Exceedingly slow rates of testing may result in creep and hence are to be avoided. Beyond the yield strength, the rate of straining shall not exceed 0.25 in. per in. per min. measured on the gage length of the specimen."

To the table in the Appendix add the following typical mechanical properties for the new alloy G2:

Tensile Strength, psi	41 000
Elongation in 2 in., per cent	7.5
Charpy Impact on Square Specimens, ft-lb	8.3

Tentative Specifications for Magnesium-Base Alloy Die Castings (**B 94 – 47 T**):[2]

Section 3.—Delete alloy AZ91 X from the table of requirements as to chemical composition and delete the footnote to this table.

Explanatory Notes.—Delete alloy AZ91 X from the table in Note 1 and the entire last paragraph from Note 2.

Add the following sentence at the end of Note 1: "In the tension testing of magnesium alloy specimens, the rate of stressing up to the yield strength shall not exceed 100,000 psi. per min. Exceedingly slow rates of testing may result in creep and hence are to be avoided.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1946 Book of A.S.T.M. Standards, Part I-B.

Beyond the yield strength, the rate of straining shall not exceed 0.25 in. per in. per min. measured on the gage length of the specimen.

Tentative Specifications for Copper-Base (Brass) Alloy Die Castings (**B 176 – 47 T**):[2]

Section 1.—Change the second sentence to read as follows: "Three brass alloy compositions are specified, designated as alloys A, B, and C.

Table I.—Add the following chemical requirements for the new alloy C:

Copper, per cent	80 to 83
Silicon, per cent	3.75 to 4.25
Lead, max., per cent	0.15
Tin, max., per cent	0.25
Manganese, max., per cent	0.15
Aluminum, max., per cent	0.15
Iron, max., per cent	0.15
Magnesium, max., per cent	0.01
Other elements, max., per cent	0.25
Zinc, per cent	remainder

Section 5.—Add the following to the note in this section: "In the tension testing of die-cast brass specimens, the rate of stressing up to the yield strength shall not exceed 100,000 psi. per min. Exceedingly slow rates of testing may result in creep and hence are to be avoided. Beyond the yield strength, the rate of straining shall not exceed 0.25 in. per in. per min. measured on the gage length of the specimen."

In this same note add to the table the following properties for the new alloy C:

Tensile strength, min., psi	90 000
Yield strength (offset = 0.2 per cent), min., psi	45 000
Elongation in 2 in. min., per cent	25

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following two standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Standard Specifications for Zinc-Base Alloy Die Castings (**B 86 – 46**):[1]

Sections 7, 8, and 9.—Delete these three sections and renumber the subsequent sections accordingly.

Appendix.—The following information is intended to supersede that contained in Sections 7, 8, and 9 and is to appear as an appendix to these specifications.

Appendix.—The data in the following table do not constitute a part of these specifications. However, they will indicate to the purchaser the mechanical properties that may be expected of tension and impact specimens when made under the best known conditions in a test bar die.

Alloy	Tension Tests on Round Specimens		Charpy Impact on Square Specimens, ft.-lb.	Brinell Hardness Number on Square Specimens, 500-kg. Load, 10-mm. Ball
	Tensile Strength, psi.	Elongation in 2 in., per cent		
XXI	46 000	4	4	90
XXIII	35 000	10	35	65
XXV	40 000	5	35	80

In the tension testing of die-cast zinc-alloy specimens the rate of straining shall not exceed 0.25 in. per in. per min. measured on the gage length of the specimen.

Standard Specifications for Lead- and Tin-Base Alloy Die Castings (**B 102 – 44**):[1]

Appendix.—Add the following statement between the second and third sentences: "In the tension testing of die-cast lead- and tin-alloy specimens, the rate of strain shall be $\frac{1}{8}$ in. per in. per min. measured on the gage length of the specimen."

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

---

[2] 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (J. J. Bowman, chairman) reported continuation of its investigation of the G2, SG2, and SG3 alloys. All of the short-time tests have been completed and there remain only the long-time atmospheric exposures. The available data appear in the appendix to this report[4]. The subcommittee wishes to express its appreciation to Messrs. G. M. Rollason, J. R. Townsend, J. C. Fox, A. E. Martin, and M. H. Jameson for the very considerable amount of work they did in planning and carrying out these tests.

The subcommittee recommended the revisions in Specifications B 85 – 46 T covered earlier in this report.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) recommended that the Standard Specifications B 86 – 46 be revised as previously noted and continued as standard.

Subcommittee IV on Physical Tests on Die Castings (E. H. Kelton, chairman) recommended that statements on speed of test in the tension testing of die-cast alloys be incorporated in all of the specifications under the jurisdiction of this committee.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) reported that specimens exposed by Committees A-5, B-3, and B-6 at Key West, Fla., had to be moved or scrapped because the land was sold to the city and was being converted into a recreation park and bathing beach. The decision was made to move the specimens to available Naval property at Fort Taylor in Key West. The moving program was directed by E. S. Taylerson at a total cost to the committee of one hundred dollars.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (V. D. Sweeney, chairman) recommended the deletion of alloy AZ91 X from Tentative Specifications B 94 – 47 T and the continuation of the specifications as tentative.

Subcommittee VIII on Brass Die-Casting Alloys (J. C. Fox, chairman) recommended that Tentative Specifications B 176 – 47 T be revised as previously noted and continued as tentative.

Subcommittee IX on Die-Casting Processes (A. E. Martin, chairman) reported that the Hoover Co. and the Bell Telephone Laboratories have built test specimen dies and that the Canada Bureau of Mines expected delivery of their die shortly.

The work of this subcommittee is handled almost exclusively by the Advisory Group and Task Groups A, B, C and D.

Task Group A on Metal and Die Temperature Studies has been inactive pending the completion and summarization of the work being done by Task Group D.

Task Group B on Studies of Pressure, Speed, and Gate Velocity has been instructed to study the measurement of plunger speeds and metal pressures in the die and to recommend suitable methods and equipment for measuring such speeds and pressures. It was also instructed to recommend a standard method for the calculation of gate velocity.

Task Group C on Studies of Gating, Dwell Time, Die Dressings and Lubricants is planning to undertake a study of the general relationship between the area of the gate and the volume and area of the casting for several general types of aluminum die castings.

Task Group D on Studies of Melting Practices is attempting to determine the quality of die castings with regard to die soldering and its mechanical properties when the alloy is melted and held at temperature for various periods of time in gas fired, crucible, reverberatory, or electric-melting equipment.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[4] See p. 191.

The castings, both as-cast and after baking at 850 F., are to be X-rayed for soundness, tested for mechanical and physical properties, analyzed for composition, and examined for gas content and surface appearance.

A Nominating Committee consisting of S. Tour, W. Sundwick, and J. J. Bowman was appointed to present at the June meeting nominees for chairman, vice-chairman, and secretary.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 89 members; 78 members returned their ballots, of whom 71 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

APPENDIX

SUMMARY REPORT OF INVESTIGATIONS OF ALUMINUM-BASE DIE-CASTING ALLOYS CONTAINING 9.5 PER CENT SILICON AND 0.5 PER CENT MAGNESIUM OR CONTAINING 8 PER CENT MAGNESIUM

At a meeting held in Philadelphia, Pa., on January 20, 1943, Subcommittee I on Aluminum-Base Die-Casting Alloys, of Committee B-6, appointed a special task group to conduct tests of aluminum-base die-casting alloys containing 9.5 per cent silicon and 0.5 per cent magnesium or containing 8 per cent magnesium. The Task Group consisted of Messrs. G. M. Rollason, J. R. Townsend, J. C. Fox, A. E. Martin, and M. H. Jameson. During the past five years, this task group has conducted a series of tests to evaluate these alloys and has reported the results to the members of Subcommittee I as they became available. The purpose of this report is to summarize the data which have been secured so far. Some long-time atmospheric exposure tests are still in progress and will be reported as they are completed.

While the investigations of the two alloys differed in some of their details, they were similar in that test bars produced by two different companies were included; also most of the laboratory tests were duplicated in two laboratories. The tests of the 8 per cent magnesium alloy included a study of the effect of holding the metal in a molten condition for 8 hr. on the magnesium content and on the original mechanical properties of the test bars, a study of the resistance to corrosion in the 20 per cent salt (sodium chloride) spray at 95 C., and of the resistance to corrosion when exposed at New York, N. Y., and at Sandy Hook, N. J.

The effect of room-temperature aging on the properties, although not a formal part of the program, was determinable from the "storage" (uncorroded) specimens tested with the bars from the various corrosion periods.

To determine the effect of the casting machine on the characteristics, both manufacturers made test bars in a gooseneck as well as in a cold chamber machine. It was interesting to note, in this connection, that the mechanical properties of the bars cast in cold chamber machines had somewhat more "scatter" than those made in the gooseneck machines.

The results of the tests of the 8 per cent magnesium alloy are summarized in Tables I to VI. Unless otherwise indicated, each mechanical property value listed is an average of five specimens. Where less than five specimens were included in the average, those omitted were ones containing a gross defect which materially reduced the properties. The impact values reported in Table III are averages of ten determinations—five from the gate end and five from the vent end of five bars.

The investigation of the 9.5 per cent silicon, 0.5 per cent magnesium alloy was planned to determine not only the properties within a few days after casting, but also the effect of room-temperature aging on the properties, since there was some indication in the literature that an alloy of this type might age-harden consider-

TABLE I.—AVERAGE CHEMICAL COMPOSITION OF TEST BARS AS DETERMINED FROM ANALYSIS SAMPLES TAKEN DURING CASTING PERIOD.

Manufacturer	Silicon, per cent	Iron, per cent	Copper, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent	Nickel, per cent	Tin, per cent
8 PER CENT MAGNESIUM ALLOY CAST IN GOOSENECK MACHINES								
A	0.21	1.00	0.05	7.85	0.03	0.06	0.00	0.00
B	0.21	1.13	0.04	7.88	0.03	0.04	0.00	0.00
8 PER CENT MAGNESIUM ALLOY CAST IN COLD CHAMBER MACHINES								
A	0.21	0.82	0.04	7.61	0.03	0.06	0.00	0.00
B	0.31	0.53	0.04	7.57	0.02	0.05	0.00	0.00
9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY, COMMERCIAL PURITY, CAST IN COLD CHAMBER MACHINES								
A	9.77	0.99	0.22	0.52	nil	nil	....	
B	9.67	1.23	0.18	0.55	0.02	0.11	0.01	0.02
9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY, HIGH PURITY, CAST IN COLD CHAMBER MACHINES								
A	9.60	0.27	0.05	0.51	nil	nil	....	
B	9.62	0.34	0.02	0.57	0.01	0.00	0.01	0.02

ably. As in the case of the 8 per cent magnesium alloy, a study of the resistance of the 9.5 per cent silicon, 0.5 per cent magnesium alloy to both salt spray and atmospheric corrosion formed an important part of the program. Some of these specimens, too, remain exposed outdoors and will be reported upon at a later date.

Since the 9.5 per cent silicon, 0.5 per cent magnesium alloy was primarily intended as an alternate for the 12 per cent silicon alloy when cold chamber machines are to be used, test bars were produced only in cold chamber machines.

The results of the tests on the 9.5 per cent silicon, 0.5 per cent magnesium alloy are reported in Table I and Tables VII to IX. As in the other tables, all mechanical properties, except impact strengths, are an average of five tests unless otherwise indicated; impact values are again averages of ten determinations.

TABLE II.—EFFECT OF HOLDING TIME AT CASTING TEMPERATURE ON MAGNESIUM CONTENT OF 8 PER CENT MAGNESIUM ALLOY.

Elapsed Time, hr.	Magnesium, Per Cent			
	Gooseneck Machine		Cold Chamber Machine	
	Manufacturer A	Manufacturer B	Manufacturer A	Manufacturer B
1	7.92	7.84	7.86	7.59
2	7.86	7.91	7.56	7.51
3	7.85	7.83	7.58	7.48
4	7.88	7.95	7.59	7.63
5	7.72	7.99	7.73	7.87
6	7.86	7.85	7.47	7.47
7	7.83	7.84	7.42	7.55
8	7.90	7.87	7.69	7.46
Average	7.85	7.88	7.61	7.57

Respectfully submitted on behalf of Subcommittee I,

J. J. BOWMAN,
Chairman.

TABLE III.—EFFECT OF HOLDING TIME AT CASTING TEMPERATURE ON THE MECHANICAL PROPERTIES OF 8 PER CENT MAGNESIUM ALLOY TEST BARS.

Elapsed Time, hr.	Manufacturer A				Manufacturer B			
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Impact Strength, ft-lb.	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Impact Strength, ft-lb.
TEST BARS CAST IN GOOSENECK MACHINES AND TESTED BY LABORATORY 1								
1	35 200	23 820	3.5	5.86	39 080	25 300	4.4	6.74
2	33 300	23 620	3.1	6.14	39 400	24 880	4.7	7.16
3	34 100	23 420	3.3	5.34	40 100	25 840	4.7	6.55
4	34 560	23 660	3.5	6.12	41 020	25 920	4.7	6.47
5	34 900	23 680	4.3	5.55	40 000	26 040	4.9	5.34
6	33 440	23 280	3.6	6.01	40 220	26 000	4.8	5.10
7	33 940	23 540	4.0	4.95	41 180	25 680	5.2	5.28
8	34 920	23 660	4.3	5.52	40 220	25 200	4.6	6.00
Average	34 295	23 585	3.7	5.68	40 153	25 608	4.8	6.08
TEST BARS CAST IN GOOSENECK MACHINES AND TESTED BY LABORATORY 2								
1	34 620	23 800	4.0	6.20	40 500	25 440	6.1	7.44
2	33 620	23 660	3.4	5.60	39 760	25 080	5.7	7.83
3	33 180	23 580	4.1	5.33	40 980	25 480	6.0	6.57
4	33 720	24 020	4.5	6.00	39 460	25 820	5.6	7.11
5	33 100	24 100	3.6	5.36	39 900	25 820	5.3	6.23
6	33 120	23 840	3.2	6.26	40 620	25 900	5.8	6.37
7	34 460	24 680	4.3	5.67	39 920	25 600	5.6	5.15
8	34 080	23 500	4.1	5.25	38 860	24 480	5.3	5.32
Average	33 738	23 898	3.9	5.71	40 000	25 453	5.7	6.50
Grand Average	34 016	23 741	3.8	5.70	40 076	25 530	5.2	6.29
TEST BARS CAST IN COLD CHAMBER MACHINES AND TESTED BY LABORATORY 1								
1	37 980	23 600	4.0	6.64	43 260	27 700	7.7	8.48
2	37 100	22 980	4.4	5.80	40 620	27 620	6.4	7.92
3	38 780	23 460	5.5	7.83	48 540	28 260	12.4	9.98
4	39 380	23 680	6.0	7.77	47 460	28 080	10.0	8.14
5	39 280	23 680	6.2	7.81	47 360	27 420	10.4	9.76
6	39 400	23 580	6.2	7.69	46 940	28 200	9.7	10.03
7	39 020	23 460	6.5	7.67	46 140	27 300	9.0	9.97
8	37 860	23 360	5.7	7.65	43 960	26 840	7.5	8.60
Average	38 600	23 475	5.6	7.36	45 535	27 678	9.1	9.11
TEST BARS CAST IN COLD CHAMBER MACHINES AND TESTED BY LABORATORY 2								
1	37 540	22 600	5.1	6.91	40 480	26 500	7.3	9.36
2	36 380	22 140	5.3	6.88	44 140	26 860	10.5	9.92
3	35 960	22 360	5.3	7.28	46 200	27 160	12.1	9.71
4	37 640	22 480	5.8	7.21	43 280	27 460	9.4	10.40
5	36 140	22 440	5.0	6.87	44 760	26 680	9.8	9.65
6	37 540	22 550[a]	5.5	6.91	42 680	26 780	8.2	9.00
7	37 700	22 240	6.1	7.93	44 440	27 200	9.4	10.20
8	36 960	22 420	5.6	7.53	43 620	25 780	9.1	9.20
Average	36 983	22 404	5.5	7.19	43 700	26 803	9.5	9.68
Grand Average	37 792	22 939	5.5	7.28	44 618	27 240	9.3	9.40

[a] Average of 4 bars.

TABLE IV.—EFFECT OF EXPOSURE TO THE 20 PER CENT SALT SPRAY SOLUTION (SODIUM CHLORIDE) AT 95 F. ON THE MECHANICAL PROPERTIES OF 8 PER CENT MAGNESIUM ALLOY TEST BARS.

Testing Laboratory	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent
TEST BARS CAST IN GOOSENECK MACHINES, EXPOSED FOR 12 WEEKS						
1	36 180	24 330	1.6	41 130	26 670	2.3
2	37 260	25 260	3.5	41 820	27 220	5.0
Average	36 720	24 795	2.5	41 475	26 945	3.6
TEST BARS CAST IN GOOSENECK MACHINES, EXPOSED FOR 26 WEEKS						
1	33 800	24 810	1.5	41 540	26 400	5.0
2	35 560	....[a]	3.0[b]	41 840	....[a]	5.4
Average	34 680	....	2.2	41 690	....	5.2
TEST BARS CAST IN GOOSENECK MACHINES, EXPOSED FOR 52 WEEKS						
1	35 030	24 270	3.0	41 540	26 800	5.0
2	36 700	24 280	3.6	41 380	26 720	4.4
Average	35 865	24 275	3.3	41 460	26 760	4.7
TEST BARS CAST IN COLD CHAMBER MACHINES, EXPOSED FOR 12 WEEKS						
1	39 520	24 530	5.2	44 390	27 600	6.6
2	37 580	24 500	4.1[b]	44 080	28 720	5.9
Average	38 550	24 515	4.6	44 235	28 160	6.2
TEST BARS CAST IN COLD CHAMBER MACHINES, EXPOSED FOR 26 WEEKS						
1	39 428[b]	24 150[b]	5.5[b]	45 758[b]	28 105[b]	8.3[b]
2	39 500	....[a]	5.2	40 780	....[a]	4.8[b]
Average	39 464	....	5.3	43 269	....	6.5
TEST BARS CAST IN COLD CHAMBER MACHINES, EXPOSED FOR 52 WEEKS						
1	39 720	24 490	6.2	46 170	28 740	6.5
2	38 160	23 920	4.5	43 720	26 940	6.7
Average	38 940	24 205	5.3	44 945	27 840	6.6

[a] Not determined through error.
[b] Average of 4 bars.

TABLE V.—EFFECT OF EXPOSURE TO THE ATMOSPHERE ON THE MECHANICAL PROPERTIES OF 8 PER CENT MAGNESIUM ALLOY TEST BARS (TESTED ONLY BY LABORATORY 1).

Exposure Period, yr.	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent
EXPOSED ON ROOF OF BELL TELEPHONE LABORATORIES, NEW YORK, N. Y.						
CAST IN GOOSENECK MACHINES						
1	36 030	24 650	3.4	40 770	26 850	4.9
2	35 740	24 720	3.0	39 920	26 570	4.7
CAST IN COLD CHAMBER MACHINES						
1	39 440	24 090	5.6	46 330[a]	28 320	9.4
2	38 520	24 090	5.2	45 050[b]	28 280	8.5[b]
EXPOSED AT SANDY HOOK, N. J.						
CAST IN GOOSENECK MACHINES						
1	36 580	25 030	3.2	42 140	27 070	4.5
CAST IN COLD CHAMBER MACHINES						
1	40 640	24 840	5.5	47 760	28 720	6.2

[a] Average of 4 bars.
[b] Average of 3 bars.

TABLE VI.—EFFECT OF AGING AT ROOM TEMPERATURE OF THE MECHANICAL PROPERTIES OF 8 PER CENT MAGNESIUM ALLOY TEST BARS.

Aging Time	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength psi.	Elongation in 2 in., per cent
CAST IN GOOSENECK MACHINES						
As cast[a]	34 020	23 740	3.8	40 080	25 530	5.2
26 months	36 300	24 950	2.8	41 230	27 030	4.3
36 months	36 380	25 050	3.5	41 900	26 720	5.0
40 months	37 220	25 060	3.3	42 780	27 600	5.2
44 months	37 160	25 360	3.2	42 540	27 700	4.2
52 months	37 820	25 240	3.6	43 280[b]	26 800[b]	5.1[b]
CAST IN COLD CHAMBER MACHINES						
As cast[a]	37 790	22 940	5.5	44 620	27 240	9.3
26 months	40 880	24 720	5.5	47 970	28 570[b]	8.4
36 months	41 880	24 970	6.0	48 200	29 110	8.8
40 months	40 480	24 880	5.4	49 300[c]	29 200[c]	10.5[c]
44 months	40 350[b]	24 750[b]	4.8[b]	47 800[b]	28 825[b]	9.9[b]
52 months	40 870[b]	25 170[b]	5.0[b]	45 750[b]	27 380[b]	7.3[b]

[a] Average of 80 bars cast over an 8-hr. period.
[b] Average of 4 bars.
[c] Average of 3 bars.

TABLE VII.—EFFECT OF EXPOSURE TO THE ATMOSPHERE ON THE MECHANICAL PROPERTIES OF 9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY TEST BARS (TESTED ONLY BY LABORATORY 1).

Exposure Period, yr.	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent
EXPOSED ON ROOF OF BELL TELEPHONE LABORATORIES, NEW YORK, N. Y.						
COMMERCIAL PURITY GRADE						
1	35 990	25 360	1.9	43 410	26 900	2.0
2	36 710	25 660	2.5	43 650	27 020	2.0
HIGH PURITY GRADE						
1	41 130	24 380	3.6	43 530	25 550	5.0
2	40 880	23 670	3.7	42 750	25 750	4.3
EXPOSED AT SANDY HOOK, N. J.						
COMMERCIAL PURITY GRADE						
1	38 860	26 150	2.0	44 030	29 320	2.9
2	39 140	25 400	2.3	43 040	29 240	1.9
HIGH PURITY GRADE						
1	42 520[a]	25 100[a]	4.0	43 410	25 630	4.0
2	42 390	25 010	3.9	43 250	25 500	3.2

[a] Average of 4 bars.

TABLE VIII.—EFFECT OF AGING AT ROOM TEMPERATURE ON THE MECHANICAL PROPERTIES OF 9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY TEST BARS CAST IN COLD CHAMBER MACHINES.

Manufacturer	Test Laboratory	Commercial Purity				High Purity			
		Tensile Stength, psi.	Yield Strength, psi.	Elongation in 2 in. per cent	Impact Strength, ft-lb.	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Impact Strength, ft-lb.
As Cast									
A	1	40 950	25 120	3.0	2.47	42 530	25 160	5.8	4.61
A	2	40 420	25 140	2.7	2.85	45 040	24 040	5.5	4.06
B	1	46 020	26 220	4.7	2.78	43 480	24 370	4.9	4.00
B	2	47 500	26 820	4.5	2.67	44 240	23 560	6.0	4.10
Average.		43 722	25 825	3.7	2.69	43 822	24 282	5.5	4.19
Aged 3 Months									
A	1	40 710	25 020	2.7	2.90	43 480	23 820	5.0	5.50
A	2	37 740	24 860	2.7	2.87	41 760	24 420	4.1	4.85
B	1	44 900	26 580	5.7	2.45	43 740	25 240	5.4	3.46
B	2	48 320	28 100	4.6	2.90	43 600	25 240	5.0	3.95
Average.		42 918	26 140	4.0	2.78	43 145	24 680	4.9	4.44
Aged 6 Months									
A	1	43 640	25 580	2.7	3.10	41 690	23 460	4.5	4.60
A	2	43 700^a	26 509^a	3.0^a	2.74	41 900	24 040	4.4	4.99
B	1	48 140	28 710	3.4	2.44	45 160	25 020	5.8	4.00
B	2	49 420	28 840	4.0	2.58	45 360	25 380	6.1	3.98
Average.		46 225	27 407	3.3	2.71	43 528	24 475	5.2	4.39
Aged 12 Months									
A	1	43 540	25 980	2.8	3.05	42 680	24 000	4.7	4.90
A	2	44 460	26 660	3.0	2.65	44 640	24 160	5.1	[illegible]
B	1	49 200	28 290	2.0	2.40	43 980	25 410	2.0	3.75
B	2	47 460	28 660	4.4	2.12	42 360	25 460	5.5	3.49
Average.		46 165	27 397	3.0	2.55	43 415	24 758	4.3	4.28
Aged 24 Months									
A	1	43 720	25 710	2.9	2.95	43 520	24 540	4.4	4.65
A	2	43 460	25 380	2.0	3.03	43 300	24 420	4.0	4.14
B	1	48 050	29 490	3.2	2.55	46 330	26 160	3.7	3.65
B	2	48 220	28 600	3.3	2.45	44 640	25 620	5.0	3.63
Average.		45 862	27 295	2.8	2.74	44 447	25 185	4.3	4.02
Aged Approximately 29 Months									
A	2	39 960	26 520	2.2	....	45 225	25 550	5.8	
B	2	47 740	30 240	3.2	....	46 180	26 480	6.2	
Average		43 850	28 380	2.7	....	45 703	26 015	6.0	
Aged Approximately 33 Months									
A	2	39 780	27 100	2.0	....	45 040	25 860	4.4	
B	2	49 540	29 520	3.7	....	45 740	25 820	5.3	
Average.		44 660	28 310	2.9	....	45 390	25 840	4.9	...
Aged Approximately 42 Months									
A	2	41 880	26 300	2.0	....	44 525^a	25 700^a	4.5^a	...
B	2	49 580	29 420	4.5	....	44 420	25 900	4.9	...
Average.		45 730	27 860	3.3	....	44 473	25 800	4.7	...

[a] Average of 4 bars.

TABLE IX.—EFFECT OF EXPOSURE TO THE 20 PER CENT SALT SPRAY SOLUTION (SODIUM CHLORIDE) AT 95 F. ON THE MECHANICAL PROPERTIES OF 9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY TEST BARS.

Testing Laboratory	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent
			COMMERCIAL PURITY, EXPOSED FOR 12 WEEKS			
1	38 120	23 370	2.5	40 490	28 480	1.1
2	37 600[a]	26 250[a]	2.0[a]	41 680	26 360	3.7
Average	37 860	24 810	2.3	41 085	27 420	2.4
			COMMERCIAL PURITY, EXPOSED FOR 26 WEEKS			
1	37 860[a]	25 360	0.9[a]	38 770	29 060	0.7
2	36 640	[b]	1.6	38 320	[b]	2.0
Average	37 250	..	1.3	38 545		1.4
			COMMERCIAL PURITY, EXPOSED FOR 52 WEEKS			
1	37 450	24 890	2.0	38 640	26 950[a]	1.8
2	31 940	23 280	1.5	33 000	26 860	1.1
Average	34 695	24 085	1.8	35 820	26 905	1.5
			HIGH PURITY, EXPOSED FOR 12 WEEKS			
1	36 180	24 330	1.6	40 530	27 150	1.0
2	39 280	25 440	3.2	37 340	27 680	2.0
Average	37 730	24 885	2.4	38 935	27 415	1.5
			HIGH PURITY, EXPOSED FOR 26 WEEKS			
1	39 610	24 190	1.7	40 490	25 230	1.4
2	41 280	[b]	3.0	40 520	[b]	3.1
Average	39 945	...	2.4	40 505	..	2.3
			HIGH PURITY, EXPOSED FOR 52 WEEKS			
1	36 830	25 010	2.2	40 330	26 790	2.6
2	40 420	24 900	3.1	38 420	24 800	2.8
Average	38 625	24 955	2.7	39 375	25 795	2.7

[a] Average of 4 bars.

[b] Not determined through error.

REPORT OF COMMITTEE B-7*
ON
LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings during the past year: one in Atlantic City, N. J. on June 17, 1947, and the other in Washington, D. C., on March 2, 1948.

During the year seven members were added to the committee and there were two removals. At the present time the committee consists of 105 members, of whom 79 are voting members; 37 are classified as producers, 30 as consumers, and 12 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, I. V. Williams.
Vice-Chairman, W. Bonsack.
Secretary, J. J. Bowman.

Committee B-7 is continuing its work of preparing a program of tests to determine the corrosion resistance of a number of aluminum and magnesium alloys. This program has advanced to the state where it is believed that a subcommittee will be set up within the next year to carry on the actual work in cooperation with Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum and Aluminum Alloy Bars, Rods and Wire (B 211 – 46 T), and
Magnesium Base Alloy Bars, Rods and Shapes (B 107 – 45 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

On October 31, 1947, the Standards Committee accepted the following recommendations:

Revision of Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 – 46 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 – 46 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 46 T).

These revised specifications also appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

I. REVISION OF TENTATIVES

The committee recommends revisions in the following six tentative specifications:

Tentative Specifications for Aluminum and Aluminum-Alloy Sheets and Plate **(B 209 – 46 T)**:[1]

Delete all references to alloys ZG43 and Clad ZG43 from Tables I and II, from the heading of Table IV, and from the table in Section 12 (*a*), since these materials are not now commercially available.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1946 Book of A.S.T.M. Standards, Part I-B.

TABLE I.—TENSILE TEST REQUIREMENTS.

Temper	Thickness, in.	Tensile Strength, psi.	Yield Strength, min., psi.	Elongation in 2 in., min., per cent
Alloy A2				
¼ Hard	0.250 to 0.500	14 000 min.	.....	9
	0.501 to 2.000	14 000 min.	.....	12
½ Hard	0.250 to 0.500	16 000 min.	.....	6
	0.501 to 1.000	16 000 min.	.....	10
Alloy M1				
¼ Hard	0.250 to 0.500	17 000 min.	.....	9
	0.501 to 2.000	17 000 min.	.....	10
½ Hard	0.250 to 0.500	19 500 min.	.....	8
	0.501 to 1.000	19 500 min.	.....	10
Alloy GR1				
¼ Hard	0.250 to 0.500	31 000 min.	.....	9
	0.501 to 2.000	31 000 min.	.....	12
½ Hard	0.250 to 0.500	34 000 min.	.....	10
	0.501 to 1.000	34 000 min.	.....	10
Alloy CG21				
Annealed	0.501 to 1.750	35 000 max.	.....	8
Heat-treated	2.001 to 3.000	56 000 min.	40 000 min.	4
Clad CG21				
Annealed	0.500 to 1.750	34 000 max.	.....	8
Heat-treated	0.500 to 1.000	62 000 min.	40 000	8
	1.001 to 1.500	60 000 min.	40 000	7
	1.501 to 2.000	60 000 min.	40 000	6
	2.001 to 3.000	56 000 min.	40 000	4
Alloy GS21				
Annealed	0.501 to 1.000	22 000 max.	.....	18
	1.001 to 3.000	22 000 max.	.....	16
Heat-treated	0.501 to 1.000	30 000 min.	16 000	18
	1.001 to 3.000	30 000 min.	16 000	16
Heat-treated and aged	0.501 to 1.000	42 000 min.	35 000	9
	1.001 to 2.000	42 000 min.	35 000	8
	2.001 to 3.000	42 000 min.	35 000	6
Clad CS41				
Heat-treated and aged	0.500 to 1.000	67 000 min.	59 000	6
Alloy ZG42				
Heat-treated and aged	2.001 to 2.500	73 000 min.	62 000	3
	2.501 to 3.000	70 000 min.	60 000	3
CLAD ZG42				
Heat-treated and aged	2.001 to 2.500	73 000 min.	62 000	3
	2.501 to 3.000	70 000 min.	60 000	3

Section 1.—In the second sentence change the number of types of alloys from "eleven" to "ten."

Section 4.—Delete this entire section and substitute therefor Section 4 of Tentative Specifications B 178 - 47 T.

Table I.—Add to this table the following chemical requirements for a new alloy:

Aluminum	remainder
Copper, max., per cent	0.20
Iron, max., per cent	0.70
Silicon, max., per cent	0.30
Manganese, per cent	1.0 to 1.5
Magnesium, per cent	0.8 to 1.3
Zinc, max., per cent	0.10
Chromium, per cent	
Titanium, per cent	
Other elements, max., per cent:	
Each	0.05
Total	0.15

Delete from Table I the columns on vanadium and nickel, per cent, as these two columns pertain only to alloys ZG43 and Clad ZG43, which are being deleted.

TABLE II.—PHYSICAL TEST REQUIREMENTS.

Temper	Thickness, in.	Tensile Strength, psi.	Elongation in 2 in., min., per cent	Bend Diameter Factor, N
Annealed	0.013 to 0.019	29 000 max.	10	0^c
	0.020 to 0.031	29 000 max.	14	0^c
	0.032 to 0.050	29 000 max.	16	0^c
	0.051 to 0.249	29 000 max.	18	0^c
	0.250 to 3.000	29 000 max.	16	
¼ Hard	0.017 to 0.019	28 000 min.	1	0^c
	0.020 to 0.031	28 000 min.	3	1
	0.032 to 0.050	28 000 min.	4	1
	0.051 to 0.113	28 600 min.	5	2
	0.114 to 0.249	28 000 min	6	4
	0.250 to 2.000	28 000 min.	...	
½ Hard	0.013 to 0.019	32 000 min.	1	2
	0.020 to 0.050	32 000 min.	3	3
	0.051 to 0.113	32 000 min.	4	4
	0.114 to 0.249	32 000 min.	5	5
	0.250 to 1.000	32 000 min.	..	..
¾ Hard	0.013 to 0.019	35 000 min.	1	6
	0.020 to 0.031	35 000 min.	2	6
	0.032 to 0.050	35 000 min.	3	8
	0.051 to 0.162	35 000 min	4	8
Hard	0.013 to 0.019	38 000 min.	1	..
	0.020 to 0.031	38 000 min.	2	
	0.032 to 0.050	38 000 min.	3	
	0.051 to 0.128	38 000 min.	4	..

Table II.—Delete all mechanical properties and bend test requirements for as-rolled plate of alloys A2, M1, and GR1 and substitute therefor "not required."

For clad alloy CG21, annealed temper, revise the present thickness range of "0.064 to 0.500" to read "0.064 to 0.499." For the same alloy, heat-treated temper, revise the present thickness range of "0.250 to 0.500" to read "0.250 to 0.499." For clad alloy CS41, heat-treated temper, revise the present thickness range of "0.040 to 0.500" to read "0.040 to 0.499." For the same alloy, heat-treated and aged, revise the present thickness range of "0.040 to 0.500" to read "0.040 to 0.499."

Add the requirements for additional size ranges at the appropriate places as given in the accompanying Table I.

Delete footnote "e" from Table II and redesignate the subsequent footnotes accingly.

Add the physical requirements for the new alloy to Table II as given in the accompanying Table II.

Table IV.—Add a reference to the new alloy in the title of this table.

Tentative Specifications for Aluminum-Alloy Drawn Seamless Tubing (**B 210 – 46 T**):[1]

Section 4.—Delete this entire section and substitute therefor Section 4 of Tentative Specifications B 221 – 47 T

Section 11 (a).—In line 2, change "class" to "type."

Section 11 (b).—In lines 2 and 6, change "soft" to "annealed."

Section 12.—In lines 5 and 6, delete "commercially straight," since a straightness tolerance is given in Section 11 (*b*).

Tentative Specifications for Aluminum and Aluminum-Alloy Bars, Rods, and Wire (**B 211 – 47 T**):[2]

Table II.—Change the requirements for alloy CM41, heat-treated temper, to read as follows:

Diameter or Thickness, in.	1.500 and under	1.501 and over
Tensile Strength, min., psi.	55 000	55 000
Yield Strength, min., psi.	32 000	30 000
Elongation in 2 in. or 4 × Diam., min., per cent	16	16

Section 7 (b).—Change the first sentence to read as follows by the addition of the words in italics and the deletion of those in brackets: "For material [up to] 1½ in. *or less* in diameter or thickness, when not tested in full section, the tension test specimen shall be taken from the center of the section; for material *over* 1½ in. [and over] in diameter or thickness, the specimens shall be taken from midway between the center and the surface."

Tentative Specifications for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (**B 221 – 47 T**):[2]

Section 7 (b).—Make the same change as given above for Section 7 (*b*) of Tentative Specifications B 211 – 47 T.

Table III.—Delete the present table and substitute the accompanying Table III.

Tentative Specifications for Magnesium-Base Alloy Extruded Round Tubing (**B 217 – 46 T**):

Table II.—Increase the upper limit of the wall thickness from "0.250" to read "0.500" in.

Table IV.—Delete column one on "Outside Diameter, 0.99 in. and under" and change the heading for column two to read "Outside Diameter, 2.99 in. and under."

These changes are being recommended to bring these specifications in agreement with present commercial practice.

Tentative Specifications for Magnesium-Base Alloy Bars, Rods, and Shapes (**B 107 – 47 T**):[2]

Table III.—Replace this table with the accompanying Table III.

II. EDITORIAL CHANGES IN SPECIFICATIONS

The committee recommends that all wrought magnesium specifications under its jurisdiction be editorially changed at the time of their next printing by deleting the present Section 4 on Sampling for Chemical Analysis and substituting Section 4 of Tentative Specifications B 221.

The committee also recommends that the following note be added, at the next printing, to each specification under its jurisdiction which includes tensile property requirements:

> NOTE.—The values obtained for the tensile properties covered by these specifications are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi. per min., and above the yield strength the movement per minute of the head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for specimens not having reduced sections). Care must be exercised, especially when making yield strength determinations, that the speed of testing does not exceed the ability of the strain- and load-indicating equipment to function satisfactorily.

The committee recommends that the following three specifications be continued as tentative without revision, with the exception of the editorial change covering Section 4 and the addition of a note in Specifications B 90 and B 91, as proposed above:

---

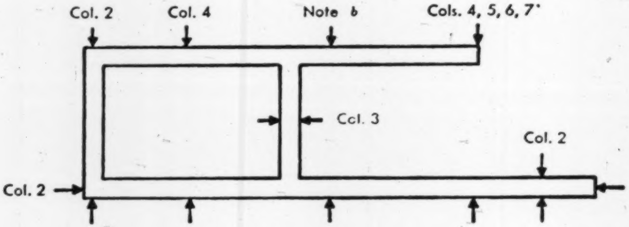
[2] 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

Tentative Specifications for:

Aluminum for Use in Iron and Steel Manufacture (B 37 – 46 T),[1]
Magnesium-Base Alloy Sheet (B 90 – 46 T),[1] and
Magnesium-Base Alloy Forgings (B 91 – 45 T).[1]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

TABLE III.—PERMISSIBLE VARIATIONS IN CROSS-SECTIONAL DIMENSIONS.



Specified Dimension, in.	Permissible Variations, plus or minus, in.[a]					
	Metal Dimensions		Space Dimensions			
	Permissible Variations from Specified Dimensions Where 75 per cent or More of the Dimension Is Metal		Permissible Variations from Specified Dimension Where More than 25 per cent of the Dimension Is Space[b c]			
	All Excepting Those Covered by Column 3	Wall Thickness[d] Completely Enclosing Space 0.11 sq. in. and Over (Eccentricity)	At Dimensioned Points ¼ to ⅝ in., excl., from Base of Leg	At Dimensioned Points ⅝ to 1¼ in., excl., from Base of Leg	At Dimensioned Points 1¼ to 2½ in., excl., from Base of Leg	At Dimensioned Points 2½ in. or more from Base of Leg
Col. 1	Col. 2	Col. 3	Col. 4	Col. 5	Col. 6	Col. 7
14.000 to 15.000, excl	0.080	10 per cent max. 0.060 min. 0.010	0.090	0.106	0.142	0.196
12.000 to 14.000, excl	0.074		0.084	0.100	0.134	0.184
10.000 to 12.000, excl	0.064		0.074	0.088	0.116	0.160
8.000 to 10.000, excl	0.054		0.064	0.074	0.100	0.136
6.000 to 8.000, excl	0.044		0.054	0.062	0.082	0.112
4.000 to 6.000, excl	0.034		0.042	0.050	0.064	0.088
2.000 to 4.000, excl	0.024		0.032	0.036	0.048	0.064
1.500 to 2.000, excl	0.016		0.024	0.028	0.034	0.050
1.000 to 1.500, excl	0.012		0.020	0.022	0.026	0.034
0.750 to 1.000, excl	0.010		0.018	0.020	0.022	0.030
0.500 to 0.750, excl	0.009		0.016	0.018	0.020	0.026
0.250 to 0.500, excl	0.008		0.014	0.016	0.018	0.022
0.125 to 0.250, excl	0.007		0.012	0.014	0.016	0.020
Under 0.125	0.006		0.010	0.012	0.014	0.016

[a] The sum of the permissible variations of component dimensions shall not be used as a permissible variation for an over-all dimension.

[b] At points less than ¼ in. from base of leg, the permissible variations in column 2 are applicable.

[c] Where the space is completely enclosed (hollow shapes), the permissible variations in column 4 are applicable.

[d] Where the dimensions specified are outside and inside, rather than the wall thickness itself, permissible variations shall be plus or minus 10 per cent of the mean wall thickness with a maximum and minimum as specified in column 3.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Aluminum-Alloy Castings (D. L. Colwell, chairman).—This subcommittee has had two task groups working on proposed revisions of Specifications B 26 and B 108 but has been unable to complete and ballot upon the changes in time for this report. It is expected, however, that the revisions will be completed by the time of the Annual Meeting and submitted to the Administrative Committee on Standards shortly thereafter.

Subcommittee III on Wrought Aluminum and Aluminum Alloys (P. V. Faragher, chairman) considered and approved the revisions to Tentative Specifications B 209, B 210, B 211, and B 221, which have been described earlier in this report. This subcommittee has in process several new specifications which it hopes to complete within the next few months.

Subcommittee IV on Magnesium and Magnesium Alloys (R. G. Thompson, chairman) recommended several changes in the tentative specifications for magnesium alloys all of which are reported under revision of tentative specifications.

Subcommittee V on Testing of Light Alloys (R. L. Templin, chairman) recommended that a paragraph on speed of testing be included in all of the product specifications under the jurisdiction of Committee B-7. This paragraph appears earlier in this report. At the March 2, 1948 meeting, Mr. A. A. Moore presented the paper "Tension Testing of Magnesium Alloy Tubing" appended hereto.[4]

The chairman appointed a task group to study the questions of frequency of sampling, type of specimen, and location of specimen in wrought products.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—During the past year the chairman distributed to members of this subcommittee reprints of the following papers contributed by members of the staff of Aluminum Research Laboratories: "Anodic Reactions of Aluminum and Its Alloys in Sulfuric and Oxalic Acid Electrolytes," by R. B. Mason and Charles J. Slunder[5] and "Measurement of Thickness of Oxide Coatings on Aluminum Alloys," by Ralph B. Mason and William C. Cochran.[6]

The subcommittee is now considering the question of whether or not it should prepare a method covering the use of the filmeter in measuring the thickness of anodic coatings.

The only other work before the subcommittee involves the testing of anodic coatings as a part of the proposed corrosion testing program which is being sponsored jointly by Committee B-7 and Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

Subcommittee VII on Codification of Light Alloys (R. B. Smith, chairman).—At the June, 1947, meeting in Atlantic City, a new code system for designating non-ferrous alloys was submitted to this subcommittee for consideration. This system was a modification of the ones used by A.S.T.M. for aluminum alloys and magnesium alloys, and was proposed to replace them. It was submitted to a letter ballot of the committee and 47 of the 96 individuals on the committee returned ballots. Twenty-four favored the new system and 15 favored continuation of the present system. As a result, the matter was referred back to Subcommittee VII for further consideration. Papers describing all of these code systems were published in the A.S.T.M. BULLETIN.[7] Temper designations for both aluminum and magnesium alloys were discussed at the March, 1948, subcommittee meeting in Washington, and progress is being made toward development of a code system for tempers.

---

[4] See p. 205.

[5] *Industrial and Engineering Chemistry*, Vol. 39, p. 1602 (December, 1947).

[6] ASTM BULLETIN, No. 148, October, 1947, p. 47.

This report has been submitted to letter ballot of the committee which consists of 79 members; 58 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

J. J. BOWMAN,
Secretary.

---

[7] R. B. Smith, "A.S.T.M. Code System for Aluminum Alloys and Magnesium Alloys," ASTM BULLETIN, No. 151, March, 1948, T.P. 58.

John C. Kiszka, "What's in an Alloy?" ASTM BULLETIN, No. 151, March, 1948, T.P. 59.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 47 T),
Aluminum-Base Alloy Sand Castings (B 26 - 47 T), and
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 47 T).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the revised tentatives appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

On December 20, 1948, the Standards Committee accepted the following recommendations submitted by Committee B 7:

New Tentative Specifications for:

Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 48 T), and
Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 - 48 T).

The new tentatives will be issued as separate reprints.

The proposed Tentative Specifications for Aluminum Alloy Extruded Tubing were accepted as tentative by the Standards Committee on December 22, 1948. The new tentative specifications have been assigned the designation B 235 - 48 T, and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

APPENDIX

TENSION TESTING OF MAGNESIUM ALLOY TUBING

By A. A. Moore[1]

The tension testing of magnesium alloy tubing is more difficult than similar tests using a machined bar in which a reduced section is provided. Any means of gripping engenders a stress raiser which causes local failure unless offset by an area of friction at the end of the plug beyond the grips. The use of a plug to prevent collapse restricts the normal reduction in size of the tube as it elongates. The change in diameter at the end of the plug causes a bending stress in the wall of the tubing in addition to the axial tensile stress.

These problems are inherent in any tension testing of tubing. Less ductile materials require more attention to testing technique. A large reduction in area offsets a loose fitting plug, and material with a high percentage of elongation is affected little by a sharp bend.

The general method of tension testing of tubing is given in the A.S.T.M. Standard Methods of Tension Testing of Metallic Materials.[2] For most commercial magnesium alloys the recommended practice is satisfactory. If relatively high strength tubing with low ductility is being tested, the following refinements in testing procedure have been found to be necessary to obtain failure in the area between the plugs.

The taper on the plug should be more gradual than shown with at least a 4 radius being used where *D* is the inside diameter of the tube. Blending this radius into the cylindrical section at the grips by means of a slight taper is also recommended. Steel or aluminum plugs are suitable for magnesium alloy tubing. A snug fit is necessary; in fact, one authority recommends a drive fit between the plug and the tubing. Care should be taken in driving the plug so that the tubing is not pre-compressed.

The usual wedge grips with coarse serrations bite deeply into the tube causing failure in the grips. Fine serrations are preferred. If finely serrated grips are not available, coarse serrations may be used if the tubing is wrapped in coarse emery cloth before gripping. No. 50 emery cloth is used on heavy tubing with a finer grade recommended for small, thin-walled tubing. The most uniform gripping is obtained with 90-deg. V grips, but the 120-deg. V grips are more commonly used. Gage marks are applied at 1-in. intervals and the elongation measured across the break.

Using the above practices, little trouble has been experienced in obtaining satisfactory tension tests on commercial alloys of magnesium tubing. When it is considered that no reduced section is used to localize failure, it is to be expected that the failures will occur at random due to either internal or external stress raisers. A small number of failures will always occur near the ends, but most of them should be between the ends of the plugs.

---

[1] The Dow Chemical Co., Midland, Mich.

[2] Standard Methods of Tension Testing of Metallic Materials (E 8 - 46), A.S.T.M. Book of Standards, Part I-A, p. 698.

REPORT OF COMMITTEE B-8*
ON
ELECTRODEPOSITED METALLIC COATINGS

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: in Atlantic City, N. J., on June 19, 1947, and in Washington, D. C., on March 5, 1948.

The committee now consists of 105 members of whom 45 are classified as producers, 11 as consumers, and 49 as general interest members.

The present officers of the committee have been re-elected for the ensuing term of two years. Mr. K. M. Huston has been elected to serve as a member-at-large of the Advisory Committee for a term of two years.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends that the standard and all tentatives under its jurisdiction be retained in their present status, pending the results of work and reviews now in progress or planned for the near future.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications, Papers, and Definitions (J. E. Starek, chairman) has appointed a working section under the chairmanship of M. R. Caldwell to make a comprehensive study of both current and proposed Government specifications and other specifications used by or acceptable to industry with a view toward revising the specifications under the jurisdiction of Committee B-8, if necessary, in the interest of standardization.

Subcommittee II on Performance Tests (H. A. Pray, chairman) has continued the exposure tests of copper-nickel-chromium deposits on steel and electrodeposited lead on steel and plans to present available data in the next annual report.

Subcommittee III on Conformance Tests (W. A. Wesley, chairman) reports progress by each of its three organized working sections. A fourth section on metal brightness tests has been organized under the chairmanship of George Jernstedt and it will start at once on the problem of evaluating the luster of metal coatings.

Subcommittee IV on Electroplating Practice (F. K. Savage, chairman) reports that its Section 1 (R. M. Wick, chairman) plans to circulate a rough draft of a recommended practice for the preparation of high-carbon steel for electroplating for criticism and comment. Section 2 (E. A. Anderson, chairman) has been organized and is initiating work toward the drafting of a recommended practice for the preparation of zinc-base die castings for electroplating.

Two more working sections were activated to prepare recommended practices for preparation for plating of copper and copper-base alloys, and of "stainless" steels.

Another group will be appointed to survey present practices regarding plating on aluminum with respect to the advisability of preparing a recommended practice on this subject.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

Subcommittee V on Supplementary Treatments for Metallic Coatings (Marc Darrin, chairman) has appointed a new working section (G. Soderberg, chairman) to review the Tentative Specifications for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (B 201 - 45 T) and to consider all chromate treatments for electrodeposited cadmium and zinc on steel. Another working section was authorized to survey present practice for phosphate-type treatments of zinc- and cadmium-plated steel.

A long-range plan for the subcommittee as a whole was outlined which includes surveys and reports on recommended practices for various supplementary treatments and special methods of testing, as well as specifications.

This report has been submitted to letter ballot of the committee, which consists of 102 voting members; 66 members returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. B. SALTONSTALL,
Secretary.

REPORT OF COMMITTEE B-9*

ON

METAL POWDERS AND METAL POWDER PRODUCTS

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1947, to June, 1948. During this time, one meeting was held in New York, N. Y., on February 19, 1948.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. A. Reich.
Vice-Chairman, W. R. Toeplitz.
Secretary, A. Squire.
Additional members of the Advisory Committee: J. E. Drapeau, Jr., B. T. duPont, F. P. Peters, and D. S. Urquhart.

The committee notes with sadness the death of A. B. Gibson, chairman of the Section on Electrical Parts, who was one of the first members of the committee and who aided very materially in lending his efforts to its formation and successful activities to date.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without change of the Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 45 T), pending revisions and additions to be made by the committee.

ADOPTION OF TENTATIVES

The committee recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Apparent Density of Metal Powders (B 212 - 46 T),
Flow Rate of Metal Powders (B 213 - 46 T),
Sieve Analysis of Granular Metal Powders (B 214 - 46 T), and
Sampling Finished Lots of Metal Powders (B 215 - 46 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman).—During the past year, this subcommittee has arrived at agreement on the 131 terms included in the glossary of terms relating to powder metallurgy.

The standard tension test bar for use by the powder metallurgy industry has been the subject of considerable subcommittee activity. Preliminary agreement has been reached on two standard bars, one to be tested in the machined condition and one in the unmachined condition. A program of testing is being drawn up to determine the suitability of these two bars for general use.

Subcommittee I will prepare technical data sheets on materials covered by the Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 45 T), and for Sintered Metal Powder Structural Parts (B 222 - 47 T). A survey of the producing and consuming industry is being made to determine what properties should be included.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M Headquarters.

Subcommittee II on Metal Powders (D. O. Noel, chairman).—A subsection on compressibility of metal powders has completed a joint program of tests involving the use of selected batches of various types of powder and different methods of lubricating the die both by including lubricant in the powder and by applying it to the punch and die surfaces. A method based on the use of oil applied to the punch and die surfaces is being prepared and will shortly be submitted to letter ballot in the main committee.

The Subsection on Methods of Chemical Analysis in cooperation with Committee E-3 on Chemical Analysis of Metals is preparing a list of existing A.S.T.M. methods applicable to metal powders. New methods relative to nonmetallic content are being considered. This work is also being carried on in cooperation with a similar subgroup of the Metal Powder Association.

A Subsection on Subsieve Particle Size Determination has continued its study of the use of the Roller air analyzer and a microscopic method for the determination of subsieve particle size. This work has not resulted in any standard methods as yet.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman): Section 1 on Bearings (D. S. Urquhart, chairman) had as its main activity during the past year the preparation of a list of standard sizes for cylindrical bearings. The preparation of such a list necessitated the establishment of uniform press fit allowances and running clearances. Substantial agreement has been achieved on these two phases. The work is proceeding on a standard size list which will be appended to Tentative Specifications B 202. The chairman of this section has been appointed to represent Committee B-9 on the special committee which is planning the proposed 1949 Bearing Symposium.

Section 2 on Structural Parts (F. V. Lenel, chairman) studied the need for specifications, particularly for brass and bronze parts in addition to Tentative Specifications B 222. A program for the correlation of the properties of structural parts made from metal powders with the properties of the anticipated standard test specimens have been put into the initial stages of planning.

Section 3 on Cemented Carbides (M. F. Judkins, chairman) has made progress during the past year on the standardization of a transverse rupture test for cemented carbides. The Advisory Committee of Committee B-9 has been considering the need for more adequate consumer representation on this section.

Section 4 on Electrical Parts (A. B. Gibson, chairman) has referred to Committee A-6 on Magnetic Properties for approval a proposed ring test for determining the magnetic properties of pole piece materials. The Advisory Committee is considering the proposal that the electrical contact activity of this section be combined in some way with the work of Committee B-4 on Electrical Heating, Resistance, and Related Alloys.

Section 5 on Friction Materials.—As noted in previous reports of Committee B-9, this section exists simply as a medium of exchange of information on friction materials with a view to engaging in standardizing work as soon as it is deemed advisable.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 39 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. REICH,
Chairman.

A. SQUIRE,
Secretary.

REPORT OF JOINT COMMITTEE*
ON
FILLER METAL

The Joint A.W.S.-A.S.T.M. Committee on Filler Metal, through its subcommittees, has continued the formulation of standards for ferrous and nonferrous filler metal. Several specifications are under consideration for revision and will be submitted for approval in the near future. Revision of one specification, revision and division of another into two separate specifications and one new specification have been submitted to the Administrative Committee on Standards. The revised specifications and the new one are indicated under the respective subcommittee responsible for them.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Deppeler.
First Vice-Chairman, O. R. Carpenter.
Second Vice-Chairman, J. H. Humberstone.
Secretary, S. A. Greenberg.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Iron and Steel Arc-Welding Electrodes (J. H. Deppeler, chairman) has devoted the past year to the revision of the Tentative Specifications for Iron and Steel Arc-Welding Electrodes (A 233 – 45 T). Meetings of the working group of this subcommittee were held on April 21 and 22, October 1, and December 11 and 12, 1947. A meeting of Subcommittee I was held on September 22, 1947. In addition to these meetings, the Technical Committee of the National Electrical Manufacturer's Association held a meeting with the working group of the Filler Metal Committee on February 14, 1947.

As a result of these meetings, it was decided to divide Tentative Specifications A 233 into two separate specifications, namely, Mild Steel Arc-Welding Electrodes[1] under the present designation and Low Alloy-Steel Arc-Welding Electrodes under a new designation (A 316 - 48 T).[1] These specifications were accepted by the Administrative Committee on Standards on June 7, 1948.

Subcommittee II on Iron and Steel Gas-Welding Rods (David Swan, chairman) held no meetings this year. Copies of the Tentative Specifications for Iron and Steel Gas-Welding Rods (A 251 – 46 T) were circulated among users in industry to determine whether any further changes are desirable. Replies indicated that the specifications are satisfactory. It is planned to revise them slightly by inclusion of a guide to welding rods similar to the guides contained in other specifications.

Subcommittee III on Aluminum and Aluminum-Alloy Filler Metal (G. O. Hoglund, chairman) has been active and is working on tentative specifications for filler metal for the various welding processes where bare filler metal is used for welding. In the interest of uniform tests for the various compositions, an attempt was made to get data on the application of guided-bend and reduced-section tension tests for welds in plate. These data were considered at a meeting in Washington and it was apparent that these testing methods could not be applied to all of the filler metal compositions, particularly the filler metal used for the welding of casting alloys.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1948 Supplement to Book of A.S.T.M. Standards, Part I-A.

It was decided to obtain further data on using test methods based on taking all-weld-metal specimens in a manner similar to that in use for testing the qualifications of brass filler metal as outlined in Navy specifications. The welded specimens have been prepared and are being tested. As soon as these data are available, a meeting of the subcommittee will be scheduled to consider the data and include the information in the specifications. The framework of the specifications has already been completed but cannot be issued to industry for comments or suggestions until the specific values for tests are developed.

Subcommittee IV on High-Alloy Steel Filler Metal (R. D. Thomas, chairman) held a meeting during the annual meeting of the American Welding Society in October, 1947, at which time it was agreed that four additional chromium-nickel alloy steels should be added in preparing a revision of the Tentative Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Welding Electrodes (A 298 - 46 T; A.W.S. A5.4 - 46 T) and that the arrangement of the specifications should be changed to parallel the new arrangement adopted for mild steel electrodes. These revised specifications[1] were accepted by the Standards Committee on August 3, 1948.

Subcommittee V on Nickel and Nickel-Alloy Filler Metal (O. B. J. Fraser, chairman) has been inactive for several years. During the forthcoming year it will either be reactivated or disbanded.

Subcommittee VI on Copper and Copper-Alloy Filler Metal (C. E. Swift, chairman).—A special group of this subcommittee met in Washington, D. C., on February 20, 1947, and again on April 25, 1947, to redraft the proposed Tentative Specifications for Copper and Copper-Alloy Metal Arc-Welding Electrodes. These specifications have been completed and were accepted as tentative by the Standards Committee on August 3, 1948, under the A.S.T.M. Designation B 225 - 48 T.[2]

Subcommittee VIII on Brazing Materials (J. L. Christie, chairman) held its first meeting on June 27, 1947, at which plans were made for the preparation of specifications covering all brazing alloys. Detailed work has been assigned to several small groups from which recommendations should be forthcoming in the near future.

Subcommittee X on Classification Designation of Filler Metal (R. David Thomas, Jr., chairman) held one meeting during the year at which the basic system for designating filler metals was presented but was deferred temporarily to consider other possible systems. A subgroup was appointed to devise other systems and this group has recently submitted its report which will be presented to the full subcommittee in the near future.

This report has been submitted to letter ballot of the committee, which consists of 67 members; 47 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. DEPPELER,
Chairman.

S. A. GREENBERG,
Secretary.

---

[2] 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.

REPORT OF JOINT COMMITTEE*
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

During the past year, the Joint Committee on Effect of Temperature on the Properties of Metals of The American Society of Mechanical Engineers and the American Society for Testing Materials has held one meeting, on December 5, 1947, in Atlantic City, N. J., during the Annual Meeting of the A.S.M.E. A second meeting was held on June 23, 1948, at Detroit, Mich., during the Annual Meeting of the A.S.T.M.

In addition, the committee sponsored a technical session on December 5, 1947, at the Annual Meeting of the A.S.M.E. at Atlantic City, at which the following paper and reports[1] were presented:

"Some 1000 F. Steam Pipe Materials," by Ernest L. Robinson.

"The Structural Stability of Several Cast Low Alloy Steels at Elevated Temperatures (Report on Project)," by V. T. Malcolm and S. Low.

"A Study of the Properties of 0.5 per cent Chromium, 0.5 per cent Molybdenum Pipe Materials (Report on Project)," by R. C. Fitzgerald, A. B. Wilder, G. V. Smith and A. E. White.

"Further Investigation on the Graphitization of Piping, for the E.E.I. and A.E.I.C., (Report on Project)," by A. M. Hall and S. L. Hoyt.

During the Annual Meeting of the A.S.T.M. the committee sponsored a session on June 23, 1948, at which the following two reports and one paper were presented:

"High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing,"[2] by J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons.

"High Temperature Bolting Materials" (Report on Project No. 16), by E. L. Robinson (Appendix I to this report).

"Studies on Graphitization Susceptibility of Carbon-Molybdenum and Chromium-Molybdenum Steels," (Report on Project 29) by J. J. Kanter and E. A. Sticha (Appendix II to this report).

Finances:

In the 1947 Report[3] of this committee, a balance of $12,520.24 was reported. Interest in the amount of $61.28 was received during the year. There were no expenditures which leaves a balance on hand of $12,581.52.

Organization and Membership:

During the year, one member has resigned and three new members have been added to the committee.

Dean C. E. MacQuigg of Ohio State University has resigned after 22 years of service on the committee. R. H. Tingey, Chief Engineer, Ship Building Division, Bethlehem Steel Co.; C. T. Evans, Jr., Metallurgical Engineer, the Elliott Co.; and Herman Weisberg, Public Service Electric and Gas Co. have been added as new members.

The committee is at present organizing sections to study more effectively the needs of industry along those lines described in the 1947 Report.[3]

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] *Transactions*, Am. Soc. Mechanical Engrs. 1947.

[2] See p. 555.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 205 (1947).

Research Projects:

Project No. 16 on Bolting Materials.—This work has been covered by a final report by E. L. Robinson which appears in Appendix I.

Project No. 18 on Effect of Manufacturing Variables on the Creep Properties of Steel (H. C. Cross, chairman).—Further work on this older project, which was discontinued during the war, is being initiated.

Project No. 29 on Stability of Steels as Affected by Temperature (J. J. Kanter, chairman).—A report on this project appears in Appendix II.

Some years ago the Joint Committee carried out a survey and prepared an index of all high-temperature testing equipment in the country. This index was used to good advantage by several Government agencies during the war. The Joint Committee at its meeting on December 5, 1947, initiated a project to resurvey the country in this particular field and to replace eventually the old index with a new one that will be up to date and which will contain more complete information. This work is well under way at this time, under the chairmanship of Mr. R. F. Miller.

Respectfully submitted on behalf of the Joint Committee,

N. L. MOCHEL,
Chairman.

H. C. CROSS,
Secretary.

APPENDIX I

HIGH-TEMPERATURE BOLTING MATERIALS[1] *

(PROJECT NO. 16)

PREPARED BY ERNEST L. ROBINSON[2]

This report prepared by the contributors[3] to Project 16 of the A.S.M.E.-A.S.T.M. Joint Committee on the Effect of Temperature on the Properties of Metals, presents the relaxation or bolting strength of a series of materials ranging from plain carbon steel to alloys suitable for bolting flanges at metal temperatures of 1000 to 1500 F. The first Progress Report[4] on Project 16 was presented at the Annual Meeting of The American Society of Mechanical Engineers in December, 1938. The report discussed bolting behavior in general and described methods and procedure for running relaxation tests. A simple formulation to describe the behavior of bolts over long periods of time was presented together with a number of typical test results showing that different constants must usually be used to describe strain relief during an anneal from those used to describe thousands of hours of service.

However, the idea of a "residual stress" after a stated period of time is a characteristic easily associated with creep strength and best determined by a relaxation test under conditions approaching conditions of service. For a full discussion of this subject, the reader is referred to the former report.

The present report collects together the performance data on a series of materials suitable for use at various temperatures from room to 1500 F. Some of the information is new and previously unpublished. But useful results that help to complete the list have been included even though they have already appeared elsewhere.

Data on high-temperature performance are set forth in Part (*a*) of Tables I, II, and III, based on the equation[5] given in the first Progress Report:[4]

$$(n-1)\, t\, r_o\, E\, S^{(n-1)} = b\, S_o^{n}$$

where:

n = the percentage increase in creep rate per percentage increase in stress. This is the slope (reciprocal) of the line on the log-log plot of stress *versus* rate of creep,

S = the residual stress in pounds per square inch after an elapsed time t in hours,

S_o = the creep strength in pounds per square inch for a nominal creep rate r_o,

E = the modulus of elasticity in pounds per square inch, and

b = the ratio of the total elasticity to that of the bar or bolt which is subject to creep.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Second and Final Report of the Subcommittee for Project 16 of the A.S.M.E.-A.S.T.M. Joint Committee on the Effect of Temperature on the Properties of Metals.

[2] Structural Engineer, Turbine Engineering Divs., General Electric Co. Fellow A.S.M.E.

[3] The original contributors to Project 16 were: Ernest L. Robinson, General Electric Co.; N. L. Mochel, Westinghouse Electric Co.; J. J. Kanter, Crane Co.; P. E. McKinney, Bethlehem Steel Co.; H. J. Kerr, Babcock and Wilcox Co.; and F. H. Norton, Massachusetts Institute of Technology. Messrs. McKinney and Kerr are no longer living.

Additional contributors to the present report include: H. C. Cross, Battelle Memorial Inst.; C. L. Clark, Timken Steel and Tube Co.; J. W. Freeman, University of Michigan; P. Cassidy, Babcock and Wilcox Co.; C. A. Crawford, International Nickel Co.; S. Low, Chapman Valve Co.; J. S. Worth, Bethlehem Steel Co.; C. T. Evans, Jr., Elliott Co.; W. C. Stewart representing the Director of U. S. Naval Engineering Experiment Station.

[4] Ernest L. Robinson, "The Resistance to Relaxation of Materials at High Temperature," *Transactions*, Am. Soc. Mechanical Engrs., August, 1939, pp. 543–554.

[5] J. J. Kanter, "Interpretation and Use of Creep Results," *Transactions*, Am. Soc. Metals, Vol. 24, December, 1936, p. 900.

It will be noted that this equation relating the residual stress S to any particular time, t, is a straight line whose slope (reciprocal) on a log-log plot of stress *versus* time is $(n - 1)$. Consequently the strength characteristics of each material may be visualized by a stress-time line on log-log paper as well as by the usual log-log plot of stress *versus* creep rate and either set of characteristics may be derived from the other by means of the equation.

TABLE I.—MODERATE TEMPERATURE MATERIALS.

PART (*a*)—RELAXATION STRENGTH. FOR VARIOUS TEST TEMPERATURES, THE 1000- AND 10,000-HR. RESIDUAL STRESSES ARE GIVEN FOR STATED INITIAL STRESSES OR LIMITING STRAINS.

	Material	Laboratory	Temperature, deg. Fahr.	Initial Strain, in. per inch	Initial Stress, psi.	Modulus of Elasticity, psi. × 10^{-6}	Creep Strength, 0.01 per cent per 1000 hr.	n	Residual Stress 1000 hr.	Residual Stress 10,000 hr.	Test Duration, hr.
No. 1	Annealed 0.35 C. steel "K-20"	G.E.	850	0.0015	30 000	20.0	3 700	4.7	3 170	1 700	
No. 2	Annealed 0.35 C. steel "K-20"	Crane	850	0.0005	10 000	20.0	3 100	4.5	2 500	1 260	
No. 3	(RW)-carbon steel "K-20"	U.S.N. E.E.S.	850		15 000	20.0	3 500	3.6	3 350	1 350	1380
No. 4	0.58 C. steel, wrought	G.E.	750	0.002	54 400	27.2	10 700	5.6	10 600	6 400	
No. 5	(RX) C.-½ Mo. "K-22" first str.	U.S.N. E.E.S.	850		25 000	21.6	14 700	12.6	14 000	11 500	1320
No. 6	(RX) C.-½ Mo. "K-22" second str.	U.S.N. E.E.S.	850		35 000	21.6	19 000	15.0	18 400	15 500	1465
No. 7	(RX) C.-½ Mo. "K-22"	U.S.N. E.E.S.	1000		20 000	19.4	4 500	4.1	4 300	2 000	2376
No. 8	A193-B12 Cr Ni.	B. & W. M.I.T.	900			25.0	4 800	5.2	4 100	2 376	

PART (*b*)—IDENTIFICATION OF MODERATE TEMPERATURE MATERIALS.

	Chemical Composition, per cent						Heat Treatment	Physical Properties			
	C	Mn	Si	Cr	Mo	Ni		Tensile Strength, 1000 psi.	Proportional Limit, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 1 No. 2	0.35	0.55	0.19				Note 1	68.0	21.5[a]	36.5	62.3
No. 3	0.34	0.55	0.19			Al 0.06	Note 2	66.7	39.0	41.3	65.5
No. 4	0.58	0.86	0.18				Note 3	104.9	64.0[a]	28.6	58.3
No. 5 No. 6 No. 7	0.16	0.63	0.24		0.53		1650 F. 1⅔ hr. FC., 1200 F. 2 hr. AC.	67.5	40.0	41.0	64.5
No. 8	0.40			0.72	1	1.21	As received	125.4	95.2	23.5	62.8

NOTE 1.—Heat to 1550 F. in 2 hr., hold 1 hr., furnace cooled to 1000 F. air cooled. Reheat to 1280 F. in 4 hr., hold 2 hr., furnace cooled to 1000 F. air cooled. Cold straighten, heat to 950 F. air cooled. Repeat all except cold straightening

NOTE 2.—1650 F. 1 hr. furnace cooled to 1000 F. air cooled, 1280 F. 2 hr. furnace cooled to 1000 F. air cooled.

NOTE 3.—1650 F. oil quench, 1250 F. furnace cooled, 1290 F. furnace cooled.

[a] Elastic limit.

Part (*b*) of Tables I, II, and III gives the chemical composition, heat treatment, and room temperature physical properties of each of the several materials.

In using these tables for proportioning bolts, it is suggested that designers make note of the duration of each test in order to avoid extrapolating the results too far. It is not unreasonable to predict 10,000 hr. performance on the basis of 2000 or 4000-hr. tests, but the higher the temperature, the less justification there is for extending the line over more than one

TABLE II.—LOW-ALLOY MATERIALS FOR HIGH-TEMPERATURE SERVICE.

PART (*a*)—RELAXATION STRENGTH OF LOW ALLOY MATERIALS FOR HIGH-TEMPERATURE SERVICE. FOR VARIOUS TEST TEMPERATURES, THE 1000- AND 10,000-HR. RESIDUAL STRESSES ARE GIVEN FOR STATED INITIAL STRESSES OR LIMITING STRAINS.

(Note that the percentage of molybdenum in Reference Nos. 39 to 54, inclusive, ranges from what corresponds to grade B14 to what corresponds to the proposed new grade B16.) (The omission of No. 45 is intentional.)

	Material	Laboratory	Temperature, deg. Fahr.	Initial Strain, in. per inch	Initial Stress, psi.	Modulus of Elasticity, psi. $\times 10^{-6}$	Creep Strength, 0.01 per cent per 1000 hr.	n	Residual Stress		Test Duration, hr.
									1000 hr.	10 000 hr.	
No. 9...	(ZR) Ni Mo	U.S.N. E.E.S.	930		25 000	21.2	14 300	12.2	13 500	11 000	415
No. 10...	A193-B4 Ni Cr Mo No. 317	G.E.	842	0.00196		27.4	40 200	25.0	39 000	35 400	3 530
No. 11...	A193-B7 S.A.E. 4140	Crane	850		30 000	24.0	17 200	8.5	17 500	12 800	
No. 12...	A193-B7 S.A.E. 4140	W. Co.	850		50 000	24.0	20 700	7.8	21 500	15 350	
No. 13...	A193-B7 S.A.E. 4140	W. Co.	930		50 000	22.0	7 500	3.75	7 950	3 550	
No. 14...	A193-B7 S.A.E. 4140	Tim. Mich.	900	0.00158	37 690	25.3	9 500	6.97	9 000	6 160	
No. 15...	A193-B7 S.A.E. 4140 No. 2105	G.E.	850	0.002		24.6	24 300	10.0	25 900	20 000	3 046
No. 16...	A193-B7 S.A.E. 4140 No. 2106	G.E.	850	0.002		26.2	20 800	8.9	21 200	15 800	3 046
No. 17...	A193-B7 S.A.E. 4140 No. 2107	G.E.	850	0.002		24.9	27 000	12.2	26 300	21 500	3 046
No. 18...	A193-B7 (339X) first str.	Crane	850		30 000	22.0	13 000	9.3	13 200	10 000	1 920
No. 19...	A193-B7 (339X) second str.	Crane	850		30 000	22.0	12 500	5.4	14 300	8 600	4 900
No. 20...	A193-B7 (475)	Crane	850		30 000			18.5	21 300	18 700	1 350
No. 21...	A193-B7 (476)	Crane	930		30 000	21.4	16 500	9.7	14 200	10 900	2 450
No. 22...	A193-B7a Hi Mo No. 2077B8	G.E.	800	0.002		28.4	35 300	15.1	36 800	31 300	2 185
No. 23...	A193-B7a Hi Mo No. 2077B8.5	G.E.	850	0.002		27.0	33 500	13.6	35 500	30 500	2 377
No. 24...	A193-B7a Hi Mo No. 2077B9	G.E.	900	0.00215		23.8	20 800	8.9	21 000	15 700	3 425
No. 25...	A193-B7a Hi Mo No. 2126B9	G.E.	900	0.002		23.5	31 000	4.6	32 000	25 000	2 740
No. 26...	A193-B7a Hi Mo No. 2144B9	G.E.	900	0.002		23.9	10 200	5.95	10 500	7 450	2 279
No. 27...	A193-B7a (478)	Crane	930		30 000	21.4	17 000	6.15	18 100	11 600	2 650
No. 28...	DM-35 Timken Cr Mo	Tim. Mich.	900	0.00173	42 270	23.8	21 000	10.5	20 400	16 000	
No. 29...	DM-45 Timken Cr Mo	Tim. Mich.	900	0.00158	38 540	22.0	15 700	7.86	15 850	11 200	
No. 30...	DM-45 (RY)	U.S.N. E.E.S.	950		25 000	23.3	14 000	51.0	15 400	14 700	2 450
No. 31...	DM-45 (RY) first str.	U.S.N. E.E.S.	1000		25 000	21.0	12 000	21.0	11 000	9 800	523
No. 32...	DM-45 (RY) second str.	U.S.N. E.E.S.	1000		25 000	21.0	16 000	23.2	15 300	13 800	598
No. 33...	A193-B11 (RJ) W Cr V seminole	U.S.N. E.E.S.	850		50 000			17.4	34 500	30 000	528
No. 34...	A193-B11 (RJ) W Cr V seminole	U.S.N. E.E.S.	950		30 000	26.0	15 200	7.2	15 300	10 500	1 152
No. 35...	A193-B11 (RJ) W Cr V seminole	U.S.N. E.E.S.	1000		25 000			8.6	10 800	8 000	384
No. 36...	A193-B13 (QV) W Mo Cr supertemp.	U.S.N. E.E.S.	950		30 000						96
No. 37...	A193-B13 (QVA) W Mo Cr supertemp.	U.S.N. E.E.S.	950		30 000	24.9	17 000	6.35	19 200	12 500	1 296
No. 38...	A193-B13 W Mo Cr supertemp.	U.S.N. E.E.S.	950		45 000	24.9	18 500	6.65	20 300	13 500	2 350
No. 39...	A193-B14 (QW) Cr Mo V templex	U.S.N. E.E.S.	850		50 000	25.2	30 000	12.0	30 800	25 000	500
No. 40...	A193-B14 (QWA) Cr Mo V templex	U.S.N. E.E.S.	950		30 000	23.7	21 000	12.9	19 400	16 000	700
No. 41...	A193-B14 (QWA) Cr Mo V templex	U.S.N. E.E.S.	950		45 000	23.7	20 000	9.1	19 700	14 800	1 300
No. 42...	A193-B14 (QWA) Cr Mo V templex	U.S.N. E.E.S.	1000		30 000	21.6	14 500	7.1	14 600	10 000	2 376
No. 43...	A193-B14 Bar 1 (339)	Crane	750		50 000	25.4		50.0	48 000	47 000	720
No. 44...	A193-B14 Bar 1 (339)	Crane	850		30 000	24.8	27 000	25.0	25 500	24 000	4 200
No. 46...	A193-B14 (446) first str.	Crane	930		30 000	24.0	19 000	10.2	18 600	14 500	2 880
No. 47...	A193-B14 (446) second str.	Crane	930		30 000	24.0	20 000	10.6	19 700	15 500	1 056
No. 48...	A193-B14 (373)	Crane	1000		30 000	23.0	16 000	6.4	17 500	11 500	2 760
No. 49...	A193-B14 Cr Mo V	Crane	850		30 000	26.5	26 200	31.0	25 900	23 500	
No. 50...	A193-B14 Cr Mo V	Crane	930		28 000	25.5	21 800	13.0	20 500	17 000	
No. 51...	A193-B14 Cr Mo V No. 867	G.E.	950	0.002		27.8	22 480	7.75	29 000	17 000	2 503
No. 52...	A193-B14 Cr Mo V first str.	B. & W. M.I.T.	900		44 000	27.0	20 000	3.0	19 000	11 700	13 579
No. 53...	A193-B14 Cr Mo V second str.	B. & W. M.I.T.	900		30 000	27.0	17 000	7.0	17 000	11 600	6 700
No. 54...	A193-B14 Cr Mo V No. 810	G.E.	932	0.002		27.0	27 070	8.0	28 400	20 400	2 375

TABLE II (*Continued*).

	Material	Laboratory	Temperature, deg. Fahr.	Initial Strain, in. per inch	Initial Stress, psi.	Modulus of Elasticity, psi. × 10^{-6}	Creep Strength, 0.01 per cent per 1000 hr.	n	Residual Stress		Test Duration, hr.
									1000 hr.	10 000 hr.	
No. 55	17-22A (Timken) Cr Mo V	Tim. Mich.	900	0.00176	40 850	25.0	27 200	18.07	25 800	22 400	
No. 56	17-22A High initial stress	Tim. Mich.	1000	0.0016	35 800	23.1	21 700	8.78	22 400	16 200	
No. 57	17-22A Medium initial stress	Tim. Mich.	1000	0.0015	33 645	23.1	18 800	10.92	19 200	14 500	
No. 58	17-22A Low initial stress	Tim. Mich.	1000	0.0015	29 220	23.1	16 400	10.52	16 600	12 600	
No. 59	17-22A (4Q)	Tim. Mich.	950	0.0018	40 700	22.0	26 200	17.1	25 200	21 900	
No. 60	17-22A (Q)	Tim. Mich.	950	0.0013	29 300	22.0	21 300	22.7	20 500	18 200	
No. 61	17-22A (6Q) first str.	Tim. Mich.	950	0.0018	36 300	22.0	23 200	18.2	22 600	20 000	
No. 62	17-22A (6Q) second str.	Tim. Mich.	950	0.0018	38 700	22.0	28 000	21.9	27 000	24 000	
No. 63	17-22A (SA) first str.	U.S.N. E.E.S.	950		25 000	24.3	19 200	42.6	18 500	17 500	
No. 64	17-22A (SA) second str.	U.S.N. E.E.S.	950		25 000	24.3	27 400	92.0	23 600	23 000	
No. 65	17-22A (SA) first str.	U.S.N. E.E.S.	1000		25 000	21.6	16 600	36.7	16 000	15 000	
No. 66	17-22A (SA) second str.	U.S.N. E.E.S.	1000		25 000	21.6	17 800	48.5	17 200	16 400	
No. 67	A193-B16 (661) first str.	Crane	1000		30 000	23.0	13 000	6.7	14 000	9 300	2 640
No. 68	A193-B16 (661) second str.	Crane	1000		50 000	23.0	12 000	3.7	13 000	7 000	2 640
No. 69	Cu Si Mo steel (491)	Crane	930		30 000	19.5	13 000	4.72	15 600	8 400	3 840
No. 70	13 Cr (HT) first str.	U.S.N. E.E.S.	930		25 000	21.8			7 500		330
No. 71	13 Cr (HT) second str.	U.S.N. E.E.S.	930		25 000	21.8			8 100		168
No. 72	13 Cr (ZV)	U.S.N. E.E.S.	930		25 000	21.4	13 400	15.5	8 900	7 600	1 054

PART (*b*)—IDENTIFICATION OF LOW ALLOY MATERIALS.

	Size of Stock	Chemical Composition, per cent								Heat Treatment	Physical Properties			
		C	Mn	Si	Cr	Mo	Ni	P	S		Tensile Strength, 1000 psi.	Proportional Limit, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 9	17⅜ P. gear	0.33	0.63	0.28	0.09	0.44	2.70	0.045	0.02	As received	102.4	45.0	24.6	54.8
No 10		0.32	0.58	0.22	0.75	0.47	1.92	0.013	0.010	1750 F.-8 hr. A.C., 1200 F.-1 hr. A.C.	120.8	80.0^a	21.5	60.6
No. 11 No. 12 No. 13		0.37	0.63	0.14	0.98	0.26		0.015	0.02	(Note 1)	133.0	55.0^a	20.0	59.0
No. 14		0.40	0.70	0.21	0.97	0.19	0.15	0.016	0.022	1550 F. O.Q., drawn 1100 F. to 269 BHN	131.0	105.0	22.0	54.1
No. 15	3¾	0.42	0.78	0.23	0.89	0.18				As received	134.9	84.9^a	19.5	55.8
No. 16										1650 F. A.C., 1650 F.-8 hr. O.Q., 1155 F.-4 hr. A.C.	116.9	82.9^a	22.0	61.6
No. 17										1740 F. A.C., 1740 F.-6 hr. A.C., 1110 F.-4 hr. A.C.	107.4	71.9^a	24.0	55.2
No. 18	1									1550 F. O.Q., draw 1100 F.	Class "C" Physicals			
No. 19	1									1550 F. O.Q., draw 1100 F.	Class "C" Physicals			
No. 20	1½	0.38	0.76	0.19	1.0	0.21				Austempered 1100 F.	108.0	67.0^b	26.0	63.0
No. 21	2⅛	0.43	0.74	0.49	0.89	0.23				Nor. 1550 F, Draw 1100 F.	113.5	84.0^b	23.0	59.0
No. 22	3	0.43	0.85	0.23	0.99	0.50				1650 F.-8 hr. O.Q., 1200 F.-4 hr. A.C.	128.9	89.9^a	23.5	65.6
No. 23	3									1650 F.-8 hr. O.Q., 1200 F.-4 hr. A.C.	128.9	89.9^a	23.5	65.6
No. 24	3									1650 F.-8 hr. O.Q., 1200 F.-4 hr. A.C.	128.9	89.9^a	23.5	65.6

[a] Elastic limit.

[b] Yield point.

(Continued on p. 218)

TABLE II (*Concluded*).

	Size of Stock	Chemical Composition, per cent								Heat Treatment	Physical Properties			
		C	Mn	Si	Cr	Mo	Ni	P	S		Tensile Strength, 1000 psi.	Proportional Limits, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 25...	3	0.43	0.85	0.23	0.99	0.50				Nor. 1650 F., 1740 F.-8 hr. A.C., 1160 F.-4 hr. A.C.	124.4	84.7[a]	17.5	57.0
No. 26...	3	0.43	0.85	0.23	0.99	0.50				1740 F.-2 hr. F.C., 1160 F.-4 hr. A.C.	103.4	52.4[a]	22.5	45.0
No. 27...	¾	0.42	0.60	0.12	0.95	0.45				Nor. 1550 F., draw 1100 F.	129.2	102.5[b]	21.0	58.0
No. 28...,		0.35	0.47	0.75	1.20	0.57		0.001	0.016	1650 F. O.Q., drawn 1250 F. to 269 BHN (7-8 grain)	128.25 126.75	100.0 97.5	24.0 25.0	65.4 64.0
No. 29...		0.45	0.57	0.70	1.26	0.57		0.018	0.018	1650 F. O.Q., drawn 1250 F. to 285 BHN	139.5 134.0	100.0 100.0	22.0 22.0	54.1 55.2
No. 30...	1⅜	0.44	0.58	0.78	1.21	0.53		0.016	0.010	1725 F.-1 hr. A.C., 1200 F.-2 hr. F.C.	124.8	72.0	22.4	55.9
No. 31...	1⅜													
No. 32...	1⅜													
No. 33...	1	0.48	0.23	0.23	1.32	V.0.28	W.1.78	0.008	0.010	1650 F. O.Q., tempered 1200 F. (by (mfr.)	233.5	150.0	14.0	42.7
No. 34...	1													
No. 35...	1													
No. 36...	1	0.39	0.76	0.25	0.38	0.59	W.1.08	0.021	0.050	As received	121.75	105.0	19.0	57.0
No. 37...	⅞	0.38	0.76	0.24	0.63	0.60	W.1.19	0.023	0.020	1725 F. 1 hr. A.C., 1200 F.-2 hr. F.C.	129.1	67.5	21.8	57.9
No. 38...	⅞													
No. 39...	1	0.38	0.66	0.22	0.96	0.49	V.0.28	0.006	0.014	As received	144.2	81.0	19.8	57.2
No. 40...	1	0.38	0.64	0.19	1.02	0.48	V.0.24	0.012	0.010	As received				
No. 41...	1	0.38	0.64	0.19	1.02	0.48	V.0.24	0.012	0.010	1725 F.-1 hr. A.C., 1200 F.-2 hr. F.C.	144.95	100.0	17.8	55.5
No. 42...	1	0.38	0.64	0.19	1.02	0.48	V.0.24	0.012	0.010	1725 F.-1 hr. A.C., 1200 F.-2 hr. F.C.	144.95	100.0	17.8	55.5
No. 43...	⅞	0.39	0.38	0.23	0.97	0.42	V.0.24			Nor. 1675., draw 1200-F.	136.7	109.2[b]	18.2	49.0
No. 44...	⅞													
No. 46...	¾	0.38	0.58	0.21	0.96	0.35	V.0.23			Nor. 1675 F., draw 1300 F.	118.7	100.25[b]	21.5	62.0
No. 47...	¾													
No. 48...	2½	0.42	0.61	0.26	1.62	0.43	V.0.14			Nor. 1675 F., draw 1200 F.	144.0	119.4[b]	20.2	59.5
No. 49...		0.35 to 0.50	0.40 to 0.70	0.15 to 0.30	0.80 to 1.10	0.30 to 0.40	V.0.2 -0.3			Nor. 1675 F., draw 1200 F.	145.0	118.0[b]	20.0	57.0
No. 50...		0.35 to 0.50	0.40 to 0.70	0.15 to 0.30	0.80 to 1.10	0.30 to 0.40	V.0.2 -0.3			Nor. 1675 F., draw 1200 F.	145.0	118.0[b]	20.0	57.0
N 51...	4	0.45	0.61	0.32	0.99	0.35	V.0.26			1650 F.-8 hr. O.Q., 1200 F.-8 hr. F.C.	131.9	95.0[a]	21.0	56.0
No. 52...	1	0.43			0.88	0.51	V.0.30			1650 F.-8 hr. O.Q., 1225 F.-8 hr. draw, 1155 F.-4 hr. draw	130.4	116.9	21.0	61.6
No. 53...	1													
No. 54...	1	0.46	0.56	0.22	0.95	0.44	V.0.22			1740 F.-8 hr. O.Q., 1200 F.-2 hr. F.C.	192.8	164.0	15.5	55.0
No. 55...		0.29	0.46	0.75	1.27	0.54	V.0.24			Nor. 1725 F., drawn 1225 F.-6 hr. to 285 BHN.	133.5	110.0	17.5	58.2
No. 56...														
No. 57...														
No. 58...														
No. 59...	4	Approx. same as (58)												
No. 60...	6													
No. 61...	6													
No. 62...	6													
No. 63...		0.29	0.48	0.75	1.22	0.50	V.0.23			1725 F.-1 hr. A.C., 1200 F.-2 hr. F.C.	143.5	87.0	14.3	55.7
No. 64...														
No. 65...														
No. 66...														
No. 67...	⅞	0.40	0.54	0.27	0.90	0.55	V.0.34			1650 F. O.Q., 1225 F. draw	141.2	125.1[b]	20.0	60.0
No. 68...														
No. 69...	1⅜	0.47	0.35	1.94	0.62	0.45	Cu 1.29			Nor. 1600 F., draw 1200 F.	140.5	105.0	20.0	43.0
No. 70...	1	0.11	0.36	0.17	12.78		0.01	0.010	0.020	1800 F.½ hr. W.Q., 1200 F.-1 hr. F.C.	98.5	56.0	23.5	71.8
No. 71...	1	0.11	0.36	0.17	12.78		0.01	0.010	0.020	1800 F. ½ hr. W.Q., 1200 F.-1 hr. F.C.	98.5	56.0	23.5	71.8
No. 72...	⅞	0.14	0.43	0.35	12.2		0.20	0.003	0.017	1800 F. ½ hr. O.Q., 1200 F.-1 hr. F.C.	115.5	60.0	22.5	69.1

NOTE 1.—1600 F. 3 hr. air cool, 1575 F. 3 hr. oil quench, 1030 F. 3 hr. furnace cool, rough machine; 1000 F. 3 hr. furnace cool.

[a] Elastic limit.

[b] Yield point.

TABLE III.—HIGH ALLOY MATERIALS.

PART (*a*)—RELAXATION STRENGTH OF HIGH ALLOY MATERIALS. FOR VARIOUS TEST TEMPERATURES, THE 1000- AND 10,000-HR. RESIDUAL STRESSES ARE GIVEN FOR STATED INITIAL STRESSES OR LIMITING STRAINS.

NOTE.—Several of the "Int. Nic." tests were run with initial strains in excess of 1 per cent. These tests were run in conventional single lever creep racks, with 10 to 1 lever ratio, and load was applied in increments of 5 lb. on the hanger. Elongation readings were made at each load increment level and the modulus of elasticity at temperature determined therefrom. All data reported by this laboratory were obtained from measured quantities of load, elongation, time, temperature, and specimen dimensions.

	Material	Laboratory	Temperature, deg. Fahr.	Initial Strain in. per in.	Initial Stress, psi	Modulus of Elasticity, psi. × 10⁻⁶	Creep Strength, 0.01 per cent per 1000 hr.	n	Residual Stress		Test Duration, hr.
									1000 hr.	10 000 hr.	
No. 73...	19-9 DL (NR46)	Battelle	1050	0.00173	28 430	22.25	23 000	21.92	22 500	20 130	340
No. 74...	19-9 DL (NR46)	Battelle	1200	0.00173	21 680	18.07	15 700	16.03	15 200	13 055	695
No. 75...	19-9 DL (NR46)	Battelle	1350	0.00173	14 990	16.26	7 000	5.425	7 000	4 115	700
No. 76...	19-9 DL (NR46)	Battelle	1500	0.00173	8 270	14.4	2 000	4.547	1 600	820	355
No. 77...	19-9 DL (Spec. No. 13)	Int. Nic.	1200	0.01232	32 000	19.9	5 200	5.172	5 000	2 850	1012
No. 78...	19-9 DL (UP) Lot 3 first str.	U.S.N. E.E.S.	1100		30 000	19.2	17 000	46.5	19 500	18 500	1752
No. 79...	19-9 DL (UP) Lot 3 second str.	U.S.N. E.E.S.	1100		30 000	19.2	25 000	26.0	23 000	21 000	475
No. 80...	19-9 W.Mo.	Int. Nic.	1200	0.01424	40 000	19.2	4 500	4.97	3 900	2 200	260
No. 81...	N-155 (N-155A1)	Battelle	1500	0.00173	15 040	15.3	4 000	7.458	3 500	2 450	725
No. 82...	N-155 (NR66)	Battelle	1500	0.00173	14 940	16.2	4 000	7.235	3 400	2 320	425
No. 83...	N-155 (UH) Lot 7	U.S.N E.E S.	1350		25 000				13 500		507
No. 84...	N-155 (UH) Lot 7 first str.	U.S.N. E.E.S.	1500		16 000	18.5			5 000		428
No. 85...	N-155 (UH) Lot 7 second str.	U.S.N. E.E.S.	1500		16 000	18.5			5 400		116
No. 86...	N-155 (UH) Lot 7 third str.	U.S.N E.E.S.	1500		16 000	18.5			7 250		260
No. 87...	N-155 (Spec. No. 14)	Int. Nic.	1200	0.0124	35 000	19.0	5 600	4.163	5 600	2 645	1012
No. 88...	422-19 cast (NR 12-78)	Battelle	1400	0.00173	20 740	16.8	7 000	6.694	6 790	4 530	
No. 89...	X-40 cast (NR 71-96)	Battelle	1400	0.00173	18 040	16.6	6 000	5.764	5 550	3 420	
No. 90...	S-816 (NR 76-73)	Battelle	1350	0.00173	20 940	21.218	12 300	9.216	11 700	8 860	310
No. 91...	S-816 (NR 76-72)	Battelle	1500	0.00173	17 240	20.665	6 100	3.679	5 700	2 355	365
No. 92...	S-816 (NR 76-80)	Battelle	1500	0.00173	15 030	20.3	4 800	3.73	4 600	1 970	730
No. 93...	S-816 (YE) Lot 2	U.S.N. E.E.S.	1350		25 000			56.5	12 800	12 300	1510
No. 94...	S-816 (YE) Lot 2	U.S.N E.E.S.	1500		15 000	20.3	7 000	12.75	6 100	5 000	3285
No. 95...	25 NT2	U.S.N. E.E.S.	1500		13 550	20.3	7 300	3.86	3 900	3 600	1679
No. 96...	Inconel "X"	Int. Nic.	1200	0.00371	77 000	22.6	42 000	9.566	49 000	37 365	1649
No. 97...	J-655 (ZM)	U.S.N. E.E.S.	1500		15 000				4 800		389
No. 98...	J-653 (ZK)	U S.N. E.E.S.	1500		15 000				6 600		225
No. 99...	6059 (ZW) first str.	U.S.N. E.E.S.	1500		15 000			44.5	3 900	3 700	964
No. 100...	6059 (ZW) second str.	U.S.N. E.E.S.	1500		15 000			35.4	4 600	4 300	2136
No. 101...	6059 (ZS)	U.S.N.	1500		15 000				6 100		569
No. 102...	E.M.E. No. 2391E0	G. E.	1000	0.0018		23.0	39 500	33.2	37 600	35 400	2745
No. 103...	16-25-6	Int. Nic.	1200	0.01245	43 000	19.5	6 100	4.91	5 900	3 200	250
No. 104...	N-153	Elliott Battelle	1200	0.00173		19.5	14 700	11.04	14 780	11 750	2000
No. 105...	N-153	Elliott Battelle	1400	0.00172		16.2	1 700	2.968	1 910	685	2000

PART (*b*)—IDENTIFICATION OF HIGH ALLOY MATERIALS.

	Size of Stock	Chemical Composition, per cent											Heat Treatment[c]	Physica Properties			
		C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	Fe	Other		Tensile Strength, 1000 psi.	Proportional Limit, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 73... No. 74... No. 75... No. 76...		0.26	0.52	0.57	18.95	9.05		1.22	1.19	0.29		Ti 0.21	Hot rolled, stress relieved 2 hr. at 1200 F.				

(Continued on p. 220)

TABLE III (*Concluded*)

	Size of Stock	Chemical Composition, per cent											Heat Treatment[c]	Physical Properties			
		C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	Fe	Other		Tensile Strength, 1000 psi.	Proportional Limit, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 77	1¼[a]	0.33	1.14	0.65	19.10	9.05		1.35	1.14	0.35		Ti 0.16	As received + 48 hr. at 1200	120.5	79.0[d]	33.5	45.8
No. 78	¾[b]	0.30	0.90	0.70	18.72	9.12	0.10	1.18	1.33	0.35	bal	Ti 0.25	2200 F. 1 hr. W.Q., 1500 F. 4 hr. F.C.	103.0	21.3	41.5	47.1
No. 79	¾[b]																
No. 80	1⅛[a]	0.10	0.64	0.51	19.41	8.25		0.32	2.08		bal		Hot rolled + 25 hr. at 1400 F. + 48 hr. at 1200 F.				
No. 81		0.15	1.46	0.42	20.84	20.58	20.48	3.18	2.56	1.06			2250 F.-1 hr. A.C., Aged 25 hr. at 1350 F.				
No. 82		0.06			20.0	20.0	20.0	3.0	2.0	1.0		N$_2$ 0.15	2250 F.-1 hr. A.C., Aged 25 hr. at 1500 F.				
No. 83	⅞[a]	0.34	0.74	0.62	20.15	21.32	20.11	2.99	2.0	1.0	bal	N$_2$ 0.17	2260 F. ½ hr. W.Q.	121.0	57.1[d]	41.0	45.9
No. 84																	
No. 85																	
No. 86																	
No. 87	1⅛[a]	0.15	1.54	0.50	21.42	19.69	20.44	3.16	2.15	0.86		N$_2$ 0.05	As received. + 2 hr. 1200 F. A.C.				
No. 88		0.40		0.51	24.75	15.92	bal	6.08			0.65		As cast, aged 50 hr. at 1350 F.				
No. 89		0.48	0.64	0.72	25.12	9.69	55.23		7.23		0.55		As cast, aged 50 hr. at 1350 F.				
No. 90		0.36	0.72	0.19	18.4	20.23	45.63	3.72	4.23	3.04	3.48		2300 F.-1 hr. W.Q., aged 50 hr. 1350 F.				
No. 91		0.36	0.72	0.19	18.4	20.23	45.63	3.72	4.23	3.04	3.48		2300 F.- 1 hr. W.Q , aged 50 hr. 1500 F				
No. 92		0.36	0.72	0.19	18.4	20.23	45.63	3.72	4.23	3.04	3.48		2300 F.- 1 hr. W.Q., aged 50 hr. 1350 F.				
No. 93		0.37	0.50	0.16	20.06	20.9	42.6	3.74	4.2	3.42	3.72		2300 F.- 1 hr. W.Q., aged 50 hr. 1500 F.				
No. 94		0.37	0.50	0.16	20.06	20.9	42.6	3.74	4.2	3.42	3.72		2300 F.- 1 hr. W.Q., aged 50 hr. 1500 F.				
No. 95		0.25	1.5	0.5	20.0	20.0	21.0	3.0	2.2		bal	Ta 2.0	2260 F. ½ hr. W.Q.				
No. 96													Hot rolled; 2100 F.-24 hr. A.C., aged 16 hr.-1300 F.				
No. 97	1[b]	0.30		0.35	23.0	23.0	25.0	4.0	1.0	1.0		N$_2$ 0.14	2260 F. ½ hr. W.Q.				
No. 98	1[b]	0.18		0.35	25.0	32.0	32.0	3.0	2.0	1.0	bal		2260 F. ½ hr. W.Q.				
No. 99	1[a]	0.18	1.0	0.40	25.0	32.0	32.0	6.5			bal		2260 F.-½ hr. W.Q., 1500F.-50 hr. A.C.				
No. 100	1[a]	0.18	1.0	0.40	25.0	32.0	32.0	6.5			bal		2260 F. ½ hr. W.Q., 1500 F.-50 hr. A.C.				
No. 101	1 1/16[b]	0.16	0.94	0.42	24.19	32.37	31.29	6.39			bal		2260 F.-½ hr. W.Q.	123.8	54.7[d]	63.0	61.6
No. 102	3[a]	0.13	0.55	0.60	19.5	11.98			3.28	1.24		N$_2$ 0.10	Cold worked at 1200 F., 1250 F.-4 hr. A.C.	117.4	69.9[d]	32.0	55.1
No. 103	1¼[a]	0.10	1.70	0.57	16.58	25.68		6.08			bal		Hot rolled + 4 hr. at 1275 F. + 48 hr. at 1200 F.				
No. 104		0.24	1.32	0.43	15.95	15.8	13.72	3.25	2.05	1.05		N$_2$ 0.14	30 min. 1500 F., 30 min. 2250 F. W.Q. 16 hr. 1400 F.				
No. 105																	

[a] Round.
[b] Square.
[c] W.Q. = water quenched, F.C. = furnace cooled, A.C. = air cooled.
[d] Yield point.

The author regrets the duplication of the last two numbers in Table III with the first two in Table IV.

square of the log paper, that is, to a life more than ten times the duration of the tests.

It is also necessary to keep in mind that bolts that perform as intended and stay tight do so because they are strong enough to resist relaxation. Initial stresses always tend to relax, and at any time before the next overhaul the residual stress should be well above the design value in order to preclude any possibility of leakage. Unlike other parts, bolts are always expected to be stressed *above* design values and to *stay* so stressed. The designer must provide large enough bolts, or in other words, choose low enough stresses so that the bolt will never relax down to the design value.

The cure for a loose bolt is always to tighten it. But if a bolt does not hold on tightening, then it is not doing its job, and a bolt of better high-temperature strength should be sought.

Noncreeping elasticity helps greatly. This may be secured by low stressed flexibility in the flange or by extra length in the bolt at somewhat reduced temperature.

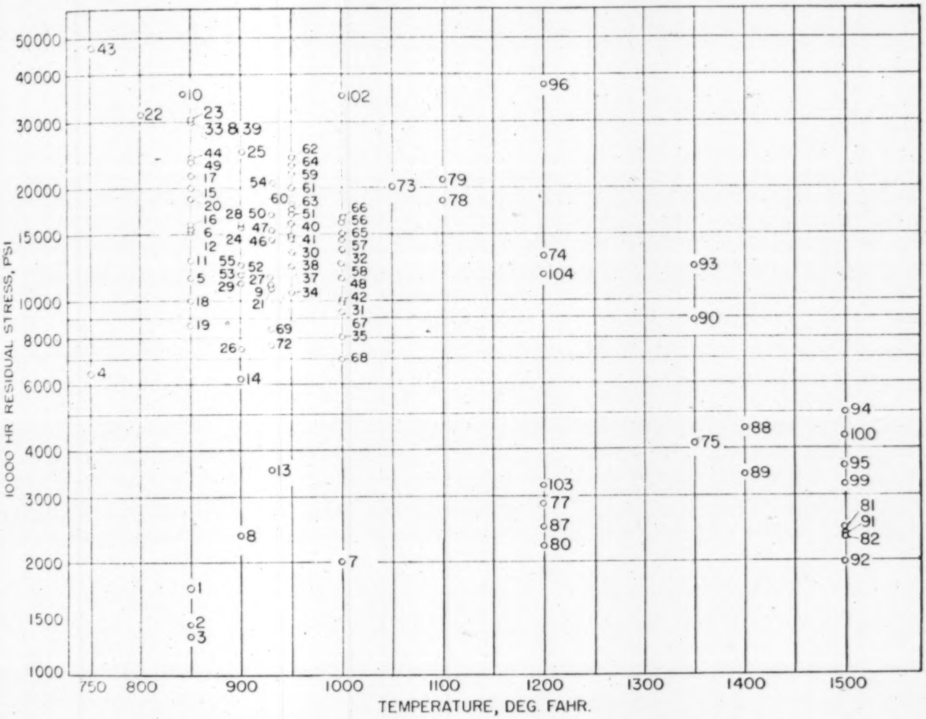


FIG. 1.—Relaxation Strength of Materials Listed in Tables I, II, and III.

This diagram shows the 10,000 hr. residual stress at temperatures ranging from 750 F. to 1500 F. for all the various materials identified by reference numbers. In making comparisons special consideration must be given to the initial stress or limiting strain. In each case the duration of the test should also be considered. (No. 55 at 900 F. is plotted incorrectly. The correct figure is 22,400. See Table II and Fig. 4.)

The materials listed in the tables fall into three groups: moderate temperature materials such as carbon and chromium-nickel steel; high-temperature ferritic materials containing tungsten, molybdenum, vanadium in various amounts with or without chromium; and the high-alloy materials which may or may not contain iron.

The high-alloy materials may have a

coefficient of thermal expansion about 50 per cent higher than in the ferritic materials. It is necessarv to keep this in mind in choosing a bolting material. Unless the bolt and the flange have approximately the same thermal expansion there is likely to be trouble.

TABLE IV.—SHORT TIME RELAXATION DATA.
(Contributed by The Chapman Valve and Mfg. Co.)

	Material	Temperature, deg. Fahr.	Initial Stress, psi.	Modulus of Elasticity, psi. × 10⁻⁶	Residual		Duration of Test, hr.
					Stress, psi.	Time, hr.	
No. 104	A193-B7a	950	60 000	23.6	30 700	100	150
No. 105	A193-B7a	800	20 000	25.4	17 000	10	15
No. 106	A193-B7a	800	30 000	25.4	23 500	10	6
No. 107	A193-B7a	800	40 000	25.4	31 000	10	15
No. 108	A193-B7a	800	60 000	25.4	48 500	10	11
No. 109	A193-B7a	950	20 000	23.6	13 000	10	10
No. 110	A193-B7a	950	30 000	23.6	21 300	10	8
No. 111	A193-B7a	950	40 000	23.6	27 700	10	9
No. 112	A193-B7a	950	60 000	23.6	43 600	10	12
No. 113	A193-B7a	1100	20 000	20.0	3 650	10	18
No. 114	A193-B7a	1100	30 000	20.0	6 700	10	15
No. 115	A193-B7a	1100	40 000	20.0	11 000	10	15
No. 116	A193-B7a	1100	60 000	20.0	24 000	10	16
No. 117	A193-B7a	1200	20 000	15.0		10	14
No. 118	A193-B7a	1200	30 000	15.0	1 000	10	16
No. 119	A193-B7a	1200	40 000	15.0	2 100	10	24
No. 120	A193-B7a	1200	60 000	15.0	15 000	10	16
No. 121	A193-B7a	950	20 000	23.6	8 500	1000	144
No. 122	A193-B7a	950	40 000	23.6	13 500	1000	144
No. 123	A193-B7a	950	60 000	23.6	18 500	1000	144
No. 124	A193-B7a	950	80 000	23.6	26 500	1000	144
No. 125	A182-F8	1000	30 000	20.5	12 750	100	144
No. 126	A182-F8	1000	30 000	20.5	8 200	100	144
No. 127	A182-F8	1200	25 000	18.7	6 200	100	144
No. 128	A182-F8	1250	25 000	18.3	6 300	100	144

	Chemical Composition, per cent								Heat Treatment	Physical Properties			
	C	Mn	Si	Cr	Mo	Ni	P	S		Tensile Strength, 1000 psi.	Proportional Limit, 1000 psi.	Elongation, per cent	Reduction of Area, per cent
No. 104–124	0.45	0.73	0.47	1.11	0.61		0.03	0.01	1450 F.-1750 F. air cool, 1250 F. draw	172.0	95.0	17.0	48.1
No 125–128	0.04	0.59	0.59	18.28		9.19	0.02	0.01	1950 F. water quench	89.0	12.0	57.0	72.5

NOTE.—All Specimens made from 1-in. round Stock.
NOTE.—Grade B7a of A.S.T.M. Specification A193 is the higher molybdenum variety of S.A.E. 4140.

Figure 1 shows the 10,000-hr. residual stresses for all the materials plotted against temperature. This diagram shows something about the spread in the reported test results and enables the selection of a suitable material for each different temperature range.

The information furnished by the various contributors enables the presentation of a number of special characteristics of relaxation behavior which are illuminating. It was the purpose of the first progress report of Project 16 to present the general nature of relaxation behavior and bolting performance. That report included numerous diagrams illustrating different variations in such behavior.

Thanks to the information contributed by various sources it is possible at this

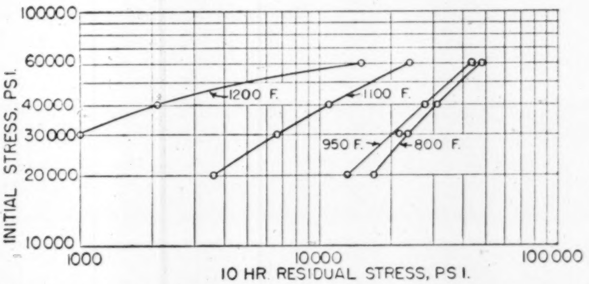


FIG. 2.—Short-Time Relaxation Data for A.S.T.M. A 193 Grade B7a Material.

The effect of initial stress on the 10-hr. residual stress as determined by The Chapman Valve and Mfg. Co.

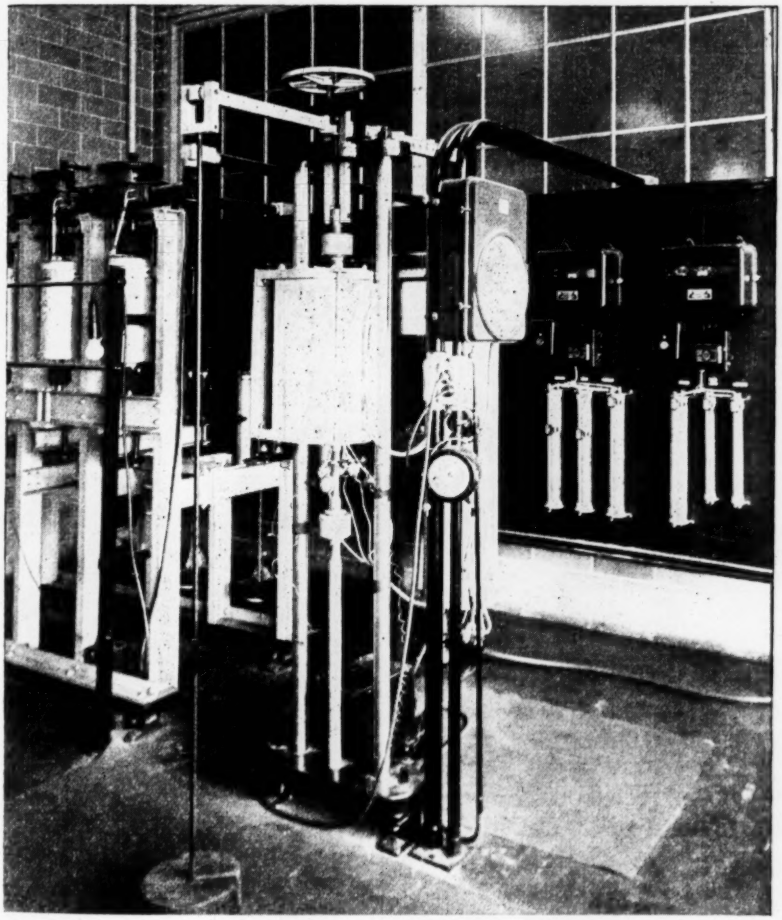


FIG. 3.—Constant Strain Automatic Relaxation Machine in the Laboratory of The Chapman Valve and Mfg. Co. Hydraulic type with recording pressure gage on weight tank.

time to give further information along these lines.

Short-time relaxation information for a variety of initial stresses has been furnished by The Chapman Valve Mfg. Co. on the high molybdenum variety of S.A.E. 4140 steel (B7*a* of A.S.T.M. Specification A 193)[6] and on 18 per cent chromium, 8 per cent nickel material (F8 of A.S.T.M. Specification A 182).[7] This information is listed in Table IV.

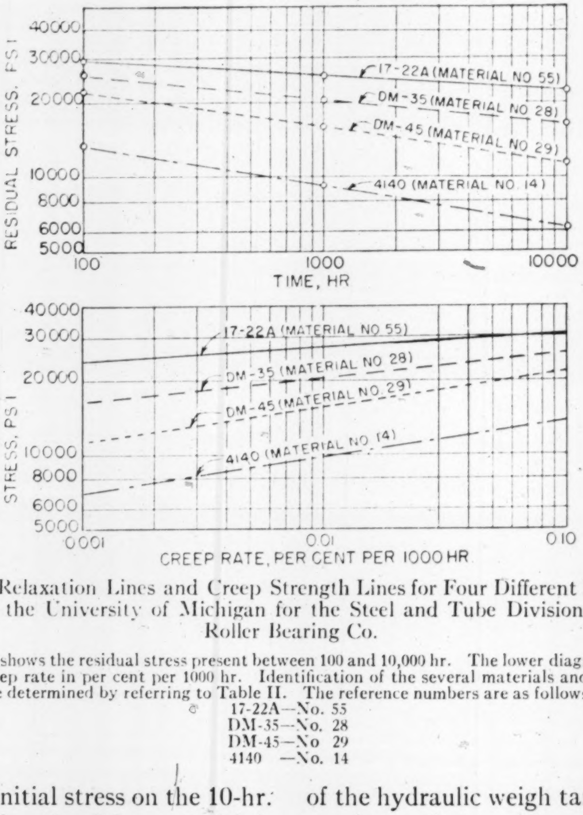


FIG. 4.—Log-Log Relaxation Lines and Creep Strength Lines for Four Different Materials at 900 F. Determined by the University of Michigan for the Steel and Tube Division of the Timken Roller Bearing Co.

The upper diagram shows the residual stress present between 100 and 10,000 hr. The lower diagram shows the relation between stress and creep rate in per cent per 1000 hr. Identification of the several materials and the initial stress and limiting strains may be determined by referring to Table II. The reference numbers are as follows:

17-22A—No. 55
DM-35—No. 28
DM-45—No 29
4140 —No. 14

The effect of initial stress on the 10-hr. residual stress for the B7*a* material is shown in Fig. 2. This diagram measures on an absolute level the effect of a 10-hr. stress relief anneal from the respective temperatures and initial stresses.

Figure 3 shows the constant-strain, automatic relaxation machine in the Research Laboratory of The Chapman Valve Mfg. Co. As the name of this machine indicates, relaxation tests run in this machine are of the constant strain type of test. This type of testing is opposed to creep testing at a constant stress.

The test bar is loaded at room temperature to the desired strain by means of the hydraulic weigh tank at the end of a primary lever. The extension of the test bar is measured by means of a conventional extensometer employing a dial gage. After loading, the test bar is brought to the desired temperature. The dial gage setting is corrected for expansion and a pair of electrical contacts built into the dial gage are set so that a change in strain equal to 0.00005 in. per inch will cause the load to decrease permitting the return of the gage to its nominal strain setting. The electrical contacts, when closed, open a solenoid valve in the weigh tank permitting the water to drain from the tank. A recording pressure gage is employed to record automatically the head of water in the weigh tank and hence the stress in the test bar.

---

[6] Tentative Specification for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 47 T), 1947 Supplement to Book of A.S.T.M. Standards, Part I-A, p. 326.

[7] Standard Specification for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 46), 1946 Book of A.S.T.M Standards, Part I-A, p. 432.

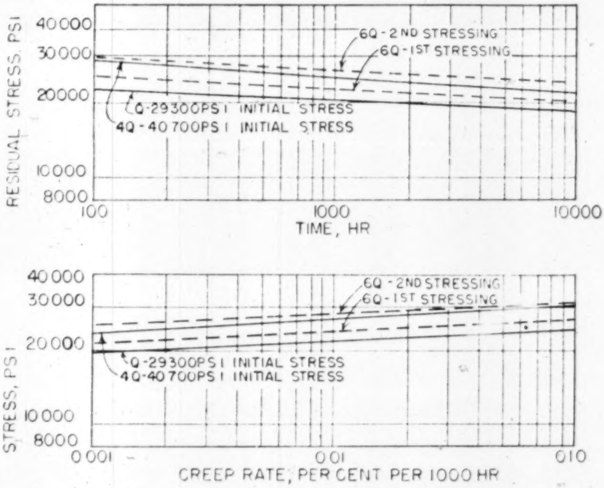


FIG. 5.—Log-Log Relaxation Lines and Creep Strength Lines at 950 F. for Material 17-22A, Nos. 59, 60, 61, and 62, as Determined by the University of Michigan for the Steel and Tube Division of the Timken Roller Bearing Co.

These lines show the effect of successive stressing and initial stress on the residual stress and creep strength.

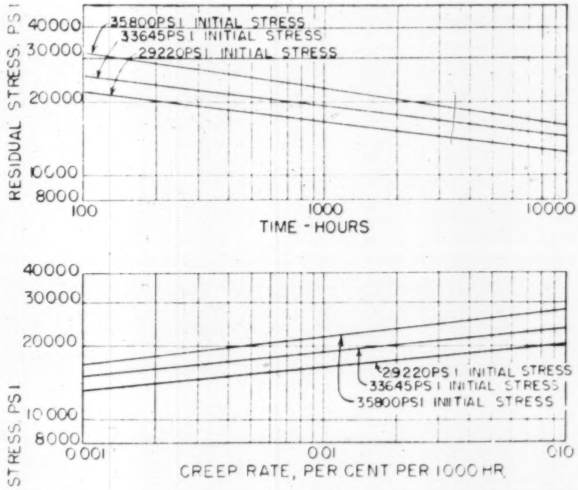


FIG. 6.—Log-Log Relaxation Lines and Creep Strength Lines at 1000 F. for Material 17-22A, Nos. 56, 57, and 58, as Determined by the University of Michigan for the Steel and Tube Division of the Timken Roller Bearing Co.

These lines show the effect of different initial stresses on the residual stress and creep strength.

The results of tests conducted at the University of Michigan and contributed by the Timken Steel and Tube Co., listed in Part (*a*) of Table II, on the effect of various initial stresses and the behavior of the material on successive tightenings are shown in Figs. 4, 5, and 6 for Timken 17-22A material. At the top of each diagram the various relaxation lines are given from which it is plain that higher initial stresses lead to higher residual stresses. In the lower half of each diagram are presented the corresponding relationships between stress and creep rate whereby the nominal creep strength as given by the relaxation tests may be determined. These diagrams plainly show the greater creep strength which characterizes the higher initial stresses. This is, of course, a measure of the effect of strain hardening as it affects the relaxation strength of the material.

The behavior illustrated both in Fig. 2 and in Fig. 6 shows that the residual stress must not be used as a quantitative measure of relaxation strength unless the initial stress or limiting strain or both are considered. For the most part, the tests reported at this time have been run under conditions approximating bolting service and therefore are roughly applicable. However, comparisons between one material and another or between one test and another should consider the limiting strain or initial stress; the higher this value, the higher will be the residual stress. Tests are usually run at 0.1 to 0.2 per cent limiting strain and are likely to be spoken of as starting with initial stresses of 30,000 psi. to 60,000 psi., these figures being obtained by multiplying the cold modulus of elasticity by the limiting strain. If the material is pulled out more than this, the resulting residual stress is likely to be higher than will occur in service unless the bolt is tightened more than desirable. On the other hand if a test is run with an initial stress of only 10,000 psi., the residual stress may be so low as not to show the capabilities of the material but only the fact that, as a bolt, the material was not tightened enough to stress it up to its capability. The results of such a test would, of course, be valid for predicting the performance of a lightly tightened bolt, and therefore it is proper to run such tests and to include the results in this survey. However, comparison between materials should either be on the basis of the same total extension (elastic plus plastic), or else some allowance should be made for the difference.

There is some variation in procedure as regards the manner of loading the specimens, adjusting the automatic controls, and providing for the periodic recording of stress, or strain, or both, thereafter. A bolt may be ordinarily thought of as tightened up cold to a predetermined strain which, it may possibly be assumed, determines the initial stress. If the strain is determined from the pitch of the thread and the number of flats on the side of the nut turned up, there may be a considerable uncertainty as to the initial stress because of the difficulty of estimating the compression of the flange due to its direct compression, design flexibility, slack in the gasket or washer material if present, or any combination of these influences. These uncertainties are sometimes eliminated in the case of important bolts by actual measurements of elastic extension by a special micrometer. In a testing machine there may or may not be actual provision for the measurement of the initial stress.

In some laboratories the initial stress is applied cold, after which the specimen is brought up to temperature and relaxation proceeds. This procedure has the virture of corresponding with common bolting practice. In other laboratories the specimen is brought up to temperature before being stressed. After the furnace is brought to temperature, strain readings are taken during application of load in order to effect a determination of the hot modulus of elasticity. While this procedure gives additional

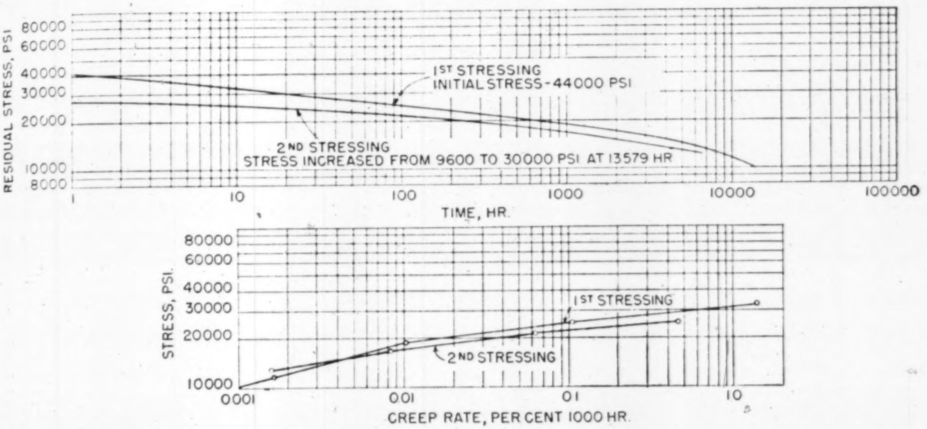


FIG. 7.—Long-Time Relaxation Test at 900 F. Run at Massachusetts Institute of Technology for the Babcock and Wilcox Co. on Material A193-B14, Nos. 52 and 53.

The upper diagram gives two relaxation lines, the upper one for the first stressing with an initial stress of 44,000 psi. and the lower line for the second stressing beginning at 30,000 psi. after 13,579 hr. during the first stressing. The lower diagram shows the relation between stress and creep rate in per cent per 1000 hr. throughout both runs. It will be noted that the stress for a particular creep rate falls off less rapidly during the second stressing than during the first stressing and that at the conclusion of the total period extending to 20,000 hr. at 900 F; the creep strength of the material was greater than during the final months under the first stressing. (This material approximates the composition under discussion for the proposed grade B16.)

TABLE V.—RESULTS OF TENSION TESTS FOR FERRITIC STEELS.

Contributed by the U. S. Naval Engineering Experiment Station. These results are descriptive of materials included in Tables I and II.

Material	Heat Treatment[a]	Test Temperature, deg. Fahr.	Tensile Strength, psi.	Yield Strength, 0.1 per cent set, psi.	Proof Stress, psi.	Proportional Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Hardness	
									Rockwell	Brinell[b]
RW Carbon Steel, A.S.M.E.-A.S.T.M. K20	1550 F. 1 hr. F.C. to 1000 F. and A.C., T. 1280 F. 2 hr., F.C. to 1000 F. and A.C.	Room	66 900	46 000	45 000	40 000	41.0	64.5	B74.4	136
			66 600	...	...	...	41.0	66.0		
			66 600	45 000	45 000	38 000	42.0	66.0		
		Avg.	66 700	45 500	45 000	39 000	41.3	65.5		
RX C-½ Mo Steel A.S.M.E.-A.S.T.M. K22	1650 F. 1⅔ hr., A.C., 1200 F. 2 hr., A.C.	Room	67 500	50 000	49 000	40 000	40.0	74.6	B76.4	140
		850	57 000	25 000	...	15 000	36.5	79.2	...	...
			57 000	24 500	...	15 000	37.0	78.8	...	...
		Avg.	57 000	24 750	...	15 000	36.7	79.0	...	...
		950	50 500	24 000	...	15 000	38.0	83.3	...	...
			51 000	24 750	...	16 000	38.0	82.5	...	...
		Avg.	50 750	24 400	...	15 500	38.0	82.9	...	...
		1050	42 750	...	...	14 500	47.0	84.7	...	...
			41 750	...	...	...	45.0	84.4	...	...
		Avg.	42 250	...	...	...	46.0	84.5	...	...
ZR Ni-Mo Steel	As received	Room	102 400	67 500	61 300	45 000	24.6	54.8	B98	228
		830	77 000	55 900	...	40 000	31.0	74.5	...	...
		930	67 000	49 900	...		32.0	82.2	...	...
		1030	53 000	42 000	...	25 000	38.0	86.2	...	...

[a] F.C. = furnace cooled, = A.C. air cooled, T = tempered.
[b] Converted from Rockwell values.

Table V continued on p. 228.

TABLE V. (*Concluded*)

Material	Heat Treatment[a]	Test Temperature, deg. Fahr.	Tensile Strength, psi.	Yield Strength, 0.1 per cent set, psi	Proof Stress, psi.	Proportional Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Hardness	
									Rockwell	Brinell[b]
Ry Cr-Mo Steel, "DM-45"	1725 F. 1 hr., A.C., 1200 F. 2 hr., F.C.	Room	130 200	90 500	87 000	79 000	19.8	58.6	B92.5	198
		Room	119 500	80 000	78 000	65 000	25.0	53.2		
		Avg.	124 850	85 250	82 500	72 000	22.4	55.9		
		650	121 750	80 600	...	58 700	21.5	64.6	...	...
		750	103 000		...		28.0	70.8	...	...
		850	101 000	71 250	...	55 000	25.0	75.0	...	...
		950	87 750	63 750	...		26.5	79.4	...	...
		1050	73 500	52 500	...	30 000	44.0	87.1	...	...
		1200	48 000	21 900	...	...	47.5	86.4	...	...
SA Cr-Mo-V Steel, "17-22A"	1725 F. 1 hr., A.C., 1200 F. 2 hr., F.C.	Room	143 500	123 000	112 500	87 000	14.3	55.7	C30.5	287
		750	122 300	97 500	...	62 500	15.5	30.7	...	...
		850	117 000	94 400	...	60 000	15.8	66.5	...	...
		950	119 800	93 700	...	58 800	18.5	67.1	...	...
		950	110 000	85 600	...	53 700	16.0	69.7	...	...
		Avg.	114 900	89 650	....	56 250	17.2	68.4	...	...
		1050	90 700	65 200	...	35 800	19.5	74.6	...	...
		1200	68 500	41 200	...	20 000	15.0	68.8	...	...
QG Cr-Mo-V Steel, A.S.T.M. B14 "Templex"	As received	Room	144 300	120 000	117 000	96 700	19.5	54.2	C32.6	302
QW Cr-Mo-V Steel, A.S.T.M. B14 "Templex"	As received	Room	144 200	116 250	103 000	81 000	19.8	57.2	C32.5	301
		850	113 700	83 250	51 000		23.0	68.7	...	...
		950	104 700	78 000	...	47 750	23.2	66.0	...	...
QWA Cr-Mo-V Steel, A.S.T.M. B14 "Templex"	1725 F. 1 hr., A.C., 1200 F. 2 hr., F.C.	Room	144 950	121 000	120 000	100 000	17.8	55.5	C31	290
		850	112 000	86 250	...	62 500	21.3	66.3	...	...
		950	98 700	79 250	...	45 000	20.7	69.9	...	...
		1000	94 500	73 500	...	...	25.0	71.4	...	...
		1100	80 500	49 400	...	...	26.0	77.3	...	...
		1200	59 000		...	...	25.5	69.8	...	...
RJ W-Cr-V Steel, A.S.T.M. B11 "Seminole"	1650 F., O.Q., T 1200 F.	Room	233 500		192 000	150 000	14.0	42.7	C45.4	435
		950	154 600	107 000	...	50 000	19.7	60.2	...	...
QV W-Mo-Cr Steel, A.S.T.M. 13 "Super Temp"	As received	Room	121 750	119 000	115 000	105 000	19.0	57.0	C22	235
		950	77 500	60 000	...	37 500	28.0	75.8	...	...
QVA W-Mo-Cr Steel, A.S.T.M. 13 "Super Temp"	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	Room	129 100	95 000	91 750	67 500	21.8	57.9	C30	283
		950	107 550	80 000	...	35 000	16.8	58.1	...	...
HT 13% Cr Steel	1800 F. ½ hr., W.Q.; 1200 F. 1 hr., F.C.	Room	98 500	75 750	68 000	56 000	23.5	71.8	B95	210
		830	78 500	63 000	...	45 000	18.0	68.8	...	...
		930	68 000	53 200	...	...	21.5	75.4	...	...
		1030	57 500	36 700	...	...	28.0	82.6	...	...
ZV 13% Cr Steel	1800 F. ½ hr., O.Q., 1200 F. 1 hr., F.C.	Room	115 500	95 850	74 400	60 000	22.5	69.1	B97	222
		830	82 900	69 500	63 700	57 300	18.0	69.3	...	...
		930	70 200	61 000	51 200	47 500	18.5	75.1	...	...
		1030	61 000	45 700	29 900	28 000	22.0	79.2	...	...

information desirable for the analysis of bolt performance, it does differ slightly from the behavior of an ordinary bolt. It is ordinarily assumed that little, if any, creep or relaxation occurs during the few minutes required for the application of the load for the determination of the elastic modulus. Sometimes care is taken to separate and record any plastic extension which may occur during loading. In other cases no such attempt is made and it is not unreasonable to assume that no serious error is involved. However, it should be noted that where no such definite segregation of effects has been made it is possible that the total elastic-plus-plastic extension or limiting strain at which a test is run may be slightly in excess of what was intended.

The tables give the initial strain or the initial stress as reported by each of the several laboratories. Where the initial strain is given, the initial stress would be equal to or possibly less than the product of the initial strain and the modulus at temperature. Where the initial stress is given, the initial strain would be equal to or more than the initial stress divided by the modulus of elasticity at temperature. The difference in the two procedures is definite but is not thought to be very great.

Another variation in procedure, the importance of which cannot be evaluated at the present time, occurs when the initial stress is lower than what corresponds to the initial strain by enough so that a considerable period of time elapses before the limiting strain is reached. This only happens with the step-down method of running a test when the creep strength of the material is slightly underestimated at the time of loading. It is likely that such a test will differ somewhat in the results yielded from a test in which the initial stress is fully up to what corresponds to the initial strain. Here again is an uncertain difference in procedure which is not thought to be of great significance.

Since the primary object of this compilation of results is to present data on the bolting strength of various materials at high temperature, it is important to emphasize that this is not a report on flange design. The tightness of bolts may be greatly prolonged by built-in elasticity provided by the designer. If such elastic follow-up is 50 to 100 F. colder than the portion of the bolt subject to relaxation, such elastic follow-up continually replaces the plastic extension, thus greatly slowing down the process of relaxation. As described in the first progress report of Project 16, tests may be run with various amounts of elastic follow-up such as three or five times the elasticity of the portion of the bolt subjected to the highest temperature. The result is to prolong the life of the joint to three to five times what it would be with a solid flange. The results presented in this compilation are all given for unit elasticity factor which corresponds to a bolt in a solid flange, in other words the most difficult type of joint to keep tight.

The results of long-time relaxation tests conducted on material B14 of A.S.T.M. Specification A 193 by the Massachusetts Institute of Technology under the sponsorship of the Babcock and Wilcox Co. are presented in Fig. 7. These tests at 900 F. extended to 13,579 hr. after the first stressing at 44,000 psi. At this time, the stress on the specimen was increased to 30,000 psi. just as if the bolt had been tightened from the 9600 psi. remaining after the first test. This test was continued 6700 hr., at which time the residual stress was 12,200 psi.

The lower half of Fig. 7 shows the actual relationship between the rate of plastic extension commonly called "creep rate" and the existing stress in the test bar. Such a creep rate is, of course, a complex total of all effects that must be taken into account in addition to the elastic relief to explain the behavior by means of a rather arbitrary but reasonably simple formula. Some degree of plastic recovery almost always occurs with relaxation and may be definitely segregated and measured when the test is run in successive steps. However, to

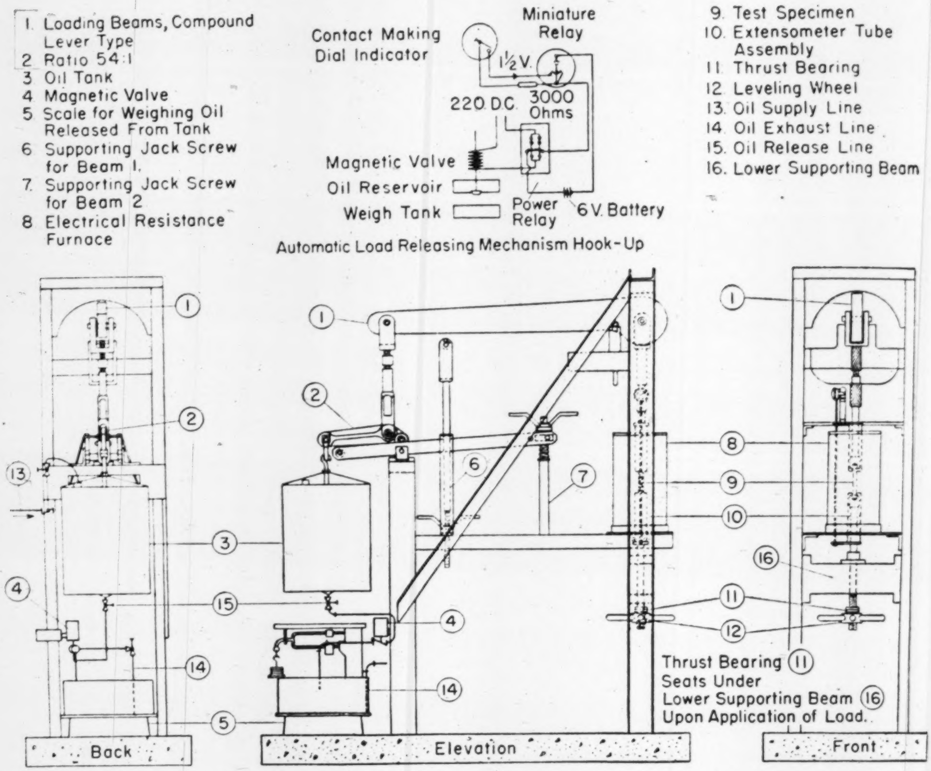

1. Loading Beams, Compound Lever Type
2. Ratio 54:1
3. Oil Tank
4. Magnetic Valve
5. Scale for Weighing Oil Released From Tank
6. Supporting Jack Screw for Beam 1.
7. Supporting Jack Screw for Beam 2
8. Electrical Resistance Furnace
Contact Making Dial Indicator
Miniature Relay
1½ V.
220 D.C.
3000 Ohms
Magnetic Valve
Oil Reservoir
Weigh Tank
Power Relay
6 V. Battery
Automatic Load Releasing Mechanism Hook-Up
9. Test Specimen
10. Extensometer Tube Assembly
11. Thrust Bearing
12. Leveling Wheel
13. Oil Supply Line
14. Oil Exhaust Line
15. Oil Release Line
16. Lower Supporting Beam
Thrust Bearing (11) Seats Under Lower Supporting Beam (16) Upon Application of Load.
Back
Elevation
Front

FIG. 8.—Details of Relaxation Test Apparatus at the U. S. Naval Engineering Experiment Station.

TABLE VI.—RESULTS OF TENSION TESTS FOR HEAT RESISTING MATERIALS.

Contributed by U. S. Naval Engineering Experiment Station. These results are descriptive of materials included in Table III.

Material	Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Strength, psi.	Yield Strength, 0.1 per cent set, psi.	Proof Stress, psi.	Proportional Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Hardness	
									Rockwell	Brinell
UP (Lot 3), 19-9DL Alloy[a]	2200 F. 1 hr., W.Q., 1500 F. 4 hr., F.C.	Room	103 000	36 500	25 000	21 300	41.5	47.1	B91.5	193
		1050	72 500	26 900	...	19 000	33.5	44.9	...	...
		1075	72 000	26 900	...	17 500	34.0	46.2	...	...
		1100	70 000	27 200	...	18 000	37.0	47.1	...	...
UH (Lot 7), N155 Alloy (0.34 C)	2260 F. ¾ hr., W.Q.	Room	121 000	57 100	...	...	41.0	45.9	B93	200
UH (Lot 3), N155 Alloy (0.35 C)	2260 F. ½ hr., W.Q.	Room	134 000	64 100	...	...	41.5	44.8	...	...
	2300 F. ¾ hr., W.Q.; 1500 F. 4 hr., F.C.	1500	62 900	37 800	...	...	7.2	6.2	...	...
YE (Lot 1), S816 Alloy (0.47 C)	2350 F. 1 hr., W.Q.	Room	156 000	62 100	...	...	27.6	24.1	B91	190
ZS 6059 Alloy (0.17 C)	2260 F. ½ hr., W.Q.	Room	123 800	54 700	...		63.0	61.6	B92.5	198

[a] Results obtained for 19-9DL alloy designated YV (Lot 2).
[b] W.Q. = water quenched, F.C. = furnace cooled.

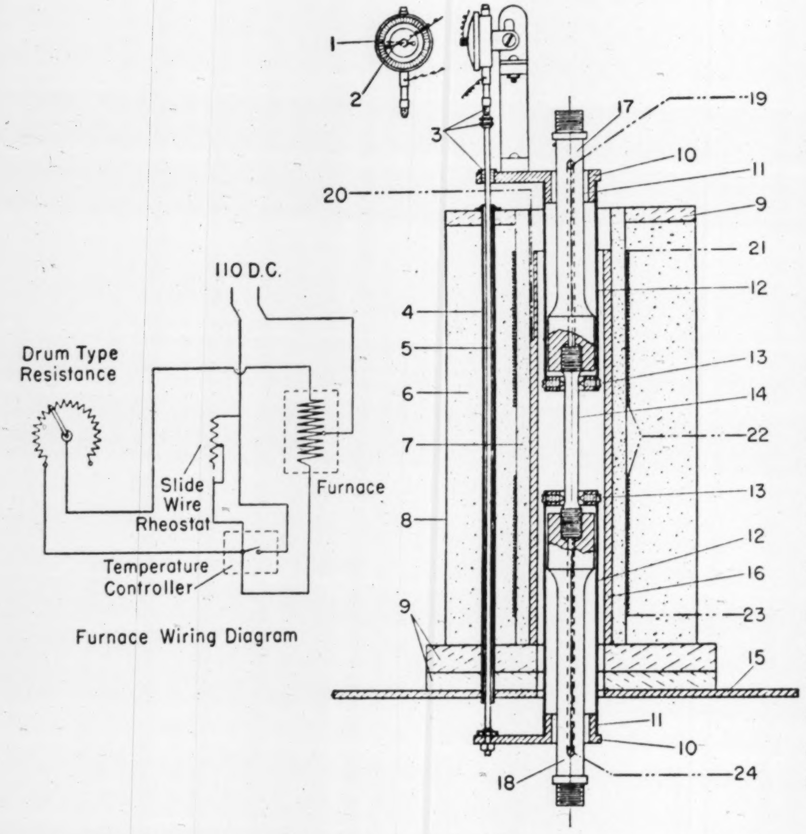
get correct results from such a simple formula, over-all behavior must be put into it. Similarly the figures given in Part (*a*) of Tables I, II, and III are intended to be used in the range between 1000 and 10,000 hr. whereas Fig. 7 makes it plain that other values would have to be used for prediction of earlier behavior. It may be noted that the nominal creep strength given in Part (*a*) of Table II for this material had decreased somewhat in the second test although there is little change in the 10,000-hr. residual stress. In fact the flatter slope of the curve represented by the higher value of n suggests that this bar may even be improving with time. Only a still longer test would have answered this question

TABLE VII.—RESULTS OF STRESS RUPTURE AND CREEP TESTS FOR FERRITIC STEELS.

Contributed by U.S. Naval Engineering Experiment Station. The 1000-hr. and 10,000-hr. residual stress figures from this Table have been included in Tables I and II.

Material	Heat Treatment^a	Temperature, deg. Fahr.	Stress for Fracture in 100, 500, and 1000 hr., psi.			Stress for Indicated minimum Creep Rate, psi.	
			100 hr.	500 hr.	1000 hr.	1% per 10 000 hr.	1% per 100 000 hr.
RW (K20) 0.35 C Steel	1550 F. 1 hr., F.C. to 1000 F. and A.C.; T 1280 F. 2 hr., F.C. to 1000 F., A.C.	850	...	...	...	8 000^{bd}	5 400^{bd}
RX (K22) C-½ Mo Steel	1650 F. 1⅔ hr., A.C.; T. 1200 F. 2 hr., A.C.	850	56 000	55 000	54 000	39 000^d	28 000^d
		950	48 000	41 000	38 000	...	...
		1000	35 700	31 500	30 000	11 000^d	7 800^d
		1050	27 000	22 000	20 300	...	...
		1100	17 000	13 000	11 300	...	...
ZR Ni-Mo Steel	As received	830	60 000	58 000	56 000	28 000^b	18 300
		930	46 000	40 000	37 500	20 800	11 000
		1030	30 400	25 000	23 000	7 000	2 500^b
		1050	27 300	23 000	21 000	...	...
RY "DM-45", Cr-Mo Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	850	96 000^b	92 000	90 000	...	...
		950	57 000	54 000	52 800	29 000	14 000
		1000	45 000^c	38 500^c	36 500^c	16 300	6 200
		1050	36 400	25 800	22 300	...	...
SA "17-22A", Cr-Mo-V Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	850	...	...	...	...	48 000
		950	98 000	96 000	95 000	...	30 000
		1000	81 000^c	69 000^c	65 000^c	27 300	19 000
		1050	66 000	47 400	41 000	...	...
QW "Templex", Cr-Mo-V Steel	As received	...	...	...	...	...	...
QWA "Templex", Cr-Mo-V Steel	As received	...	...	...	...	...	...
QWA "Templex", Cr-Mo-V Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950	75 000	65 000	61 000	40 000^b	29 000
		1000	62 000	49 000	43 500	25 600^c	17 500^c
		1050	...	...	...	15 000	9 400
RJ "Seminole", W-Cr-V Steel	O.Q. from 1650 F., T. 1200 F. by manufacturer	850	...	...	...	...	...
		950	...	...	...	...	23 000
		1000	...	...	...	28 000	16 000
QV	As received	950	...	...	...	14 800	6 800
QVA "Super Temp", W-Mo-Cr Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950	62 000	55 000	50 500	...	21 000^b
HT 13% Cr Steel	1800 F. ½ hr., W.Q.; 1200 F. 1 hr., F.C.	830	59 000	55 000	54 000	32 000^b	20 300
		930	40 000	32 300	30 400	11 700	6 500^b
		1030	23 000	19 000	17 800	2 800	...
		1050	21 800	17 300	15 700	...	...
ZV 13% Cr Steel	1800 F. ½ hr., O.Q.; 1200 F. 1 hr., F.C.	830	67 000	63 000	61 000	...	...
		930	49 000	46 000	45 000	...	13 400
		1030	34 500	30 500	28 800	...	2 400

[a] A.C. = air cooled, T. = tempered, F.C. = furnace cooled, O.Q. = oil quenched, W.Q. = water quenched.
[b] Extrapolated.
[c] Interpolated.
[d] From A.S.M.E.-A.S.T.M. report.



1-Dial indicator (0.0001 in. per division)
2-Platinum wire with carbon contact
3-Insulator
4-Metal tube
5-Invar rod
6-Diatomaceous earth
7-Refractory tube
8-Stainless steel jacket
9-Asbestos board
10-Aluminum alloy bracket
11-3 round-nose set screws 120° apart to centralize the assembly
12-Inconel tube
13-3 pointed set screws 120° apart
14-Test specimen (4-in. gage length)
15-Furnace support plate
16-Steel tube
17-Top adapter connected to lever system
18-Bottom adapter connected to anchor
19-To temperature recorder
20-Thermocouple to controller
21-To drum type rheostat
22-Connection to power line
23-Through controller to power line
24-To temperature recorder

FIG. 9.—Details of Specimen Assembly, Furnace and Extensometer for Relaxation Test in U. S Naval Engineering Experiment Station.

TABLE VIII.—RESULTS OF STRESS RUPTURE AND CREEP TESTS FOR HEAT-RESISTING MATERIALS.

Contributed by U. S. Naval Engineering Experiment Station. The 1000- and 10,000-hr. residual stress figures have been included in Table III.

Material	Heat Treatment[a]	Temperature, deg. Fahr.	Stress, for Fracture in 100, 500, and 1000 hr., psi.			Stress for Indicated minimum Creep Rate, psi.	
			100 hr.	500 hr.	1000 hr.	1% per 10 000 hr.	1% per 100 000 hr.
UP (Lot 3) 19-9DL Alloy	2200 F. 1 hr., W.Q.; 1500 F. 4 hr., F.C.	930 1100	85 000 54 000	72 000 52 000	68 000 50 000	... ...	... ...
UH (Lot 3) N155 Alloy (0.35 C)	2250 F ¾ hr., W.Q.; 1500 F. 4 hr., F.C.	1500	23 000	18 000	16 300[b]	...	...
	2300 F ¾ hr., W.Q.; 1500 F. 4 hr., F.C.	1500	17 600	15 000	14 200	12 000	9800
UH (Lot 1) N155 Alloy (0.28 C)	2200 F. 1 hr. W.Q.; 1500 F. 4 hr., F.C.	1500	20 300	17 000	15 800	...	7500
25NT-2 Alloy (0.25 C)	2260 F. ½ hr., W.Q.	1500	18 200	14 800	13 400	10 400	7300
YE (Lot 1) S816 Alloy	2350 F. 1 hr., W.Q.	1500	25 000	21 400	20 000	13 000	9600
ZS 6059 Alloy	2260 F. ½ hr., W.Q.	1500	17 500	14 000	12 800	9 400	7400

[a] W.Q. = water quenched, F.C. = furnace cooled
[b] Extrapolated.

TABLE IX.—RESULTS OF RELAXATION TESTS FOR FERRITIC STEELS.

Contributed by the U. S. Naval Engineering Experiment Station. These results are descriptive ot materials included in Tables I and II.

Material	Heat Treatment[a]	Temperature, deg. Fahr.	Initial Stress, psi.	Residual Stress, psi.					Test Duration, hr.
				100 hr.	500 hr.	1000 hr.	10 000 hr.[c]	End of Test	
RW (K-20) Carbon Steel	1550 F. 1 hr., F.C. to 1000 F. and A.C.; T. 1280 F. 2 hr., F.C. to 1000 F., A.C.	850	15 000	5 750	4 200	3 350	1 350	2 900	1380
RX (K-22) C-½ Mo Steel	1650 F. 1⅔ hr., A.C.; 1200 F. 2 hr., A.C.	850 850 1000	25 000 35 000[b] 20 000	16 800 23 000 9 250	14 500 18 800 5 300	14 000 18 400 4 300	11 500 15 500 2 000	13 750 18 250 3 680	1320 1465 2376
ZR Ni-Mo Steel	As received	930	25 000	15 100	14 700	13 500[c]	11 000	14 750	415
RY Cr-Mo Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950 1000 1000	25 000 25 000 25 000[b]	16 500 12 500 16 400	15 800 11 400 16 200	15 400 11 000[c] 15 300	14 700 9 800 13 800	15 100 11 800 16 200	2450 523 598
SA Cr-Mo-V Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950 950 1000 1000	25 000 25 000[b] 25 000 25 000[b]	19 000 23 800 16 800 17 600	18 600 23 700 16 500 17 500	18 500[c] 23 600 16 000 17 200	17 500 23 000 15 000 16 400	18 600 23 500 16 500 17 450	409 1011 692 671
QW Cr-Mo-V Steel QWA Cr-Mo-V Steel	As received As received	850 950	50 000 30 000	39 500 23 600	33 250 20 800	30 800 19 400	25 000 16 000	33 250 20 300	505 700
QWA Cr-Mo-V Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950 1000	45 000 30 000	25 700 19 200	20 100 16 000	19 700 14 600	14 800 10 000	19 500 12 950	1300 2376
RJ W-Cr-V Steel	O.Q. from 1650 F. and T. at 1200 F. by manufacturer	850 950 1000	50 000 30 000 25 000	40 000 21 000 17 300	36 200 17 200 13 500	34 500[c] 15 300 10 800[c]	30 000 10 500 8 000	36 200 15 000 14 000	528 1152 384
QV W-Mo-Cr Steel	As received	950	30 000	9 350	5 600[c]			9 350	96
QVA W-Mo-Cr Steel	1725 F. 1 hr., A.C.; 1200 F. 2 hr., F.C.	950 950	30 000 45 000	25 200 32 500	21 500 24 000	19 200 20 300	12 500 13 500	17 700 17 400	1296 2350
HT 13% Cr Steel	1800 F. ½ hr., W.Q.; 1200 F. 1 hr., F.C.	930 930	25 000 25 000[b]	8 700 9 700	7 900 8 500[c]	7 500[c] 8 100[c]		8 200 9 200	330 168
ZV 13% Cr Steel	1800 F. ½ hr., O.Q.; 1200 F. 1 hr., F.C.	930	25 000	10 300	9 300	8 900	7 600	8 900	1054

[a] F.C. = furnace cooled, A.C. = air cooled, T = tempered, W.Q. = water quenched, O.Q. = oil quenched.
[b] Same specimen as above test, reloaded.
[c] Extrapolated.

TABLE X.—RESULTS OF RELAXATION TESTS FOR HEAT-RESISTING MATERIALS.

Contributed by the U. S. Naval Engineering Experiment Station. These results are descriptive of materials included in Table III.

Material	Heat Treatment[a]	Temperature, deg. Fahr.	Initial Stress, psi.	Residual Stress, psi.					Test Duration, hr.
				100 hr.	500 hr.	1000 hr.	10 000 hr.[c]	End of Test	
UP (Lot 3) 19-9DL Alloy	2200 F. 1 hr., W.Q., 1500 F. 4 hr., F.C.	1100 1100	30 000 30 000[b]	20 800 24 100	19 800 24 000	19 500 23 000[c]	18 500 21 000	19 500 24 000	1752 475
UH (Lot 7) N155 Alloy	2260 F. ½ hr., W.Q.	1350 1500 	25 000 16 000 16 000[b] 16 000[b]	15 100 5 300 5 470 7 300	14 000 5 000 5 400[c] 7 200[c]	13 500[c] 5 000[c] 5 470[c] 7 000[c]	. . . . 	14 000 5 000 5 470 7 250	502 428 116 260
25NT-2 Alloy	2260 F. ½ hr., W.Q.	1500	13 550	4 700	4 000	3 900	3 600	3 900	1679
YE (Lot 2) S816 Alloy	2300 F. 1 hr., W.Q.; 1500 F. 50 hr., A.C.	1350 1500	25 000 15 000	13 000 8 900	12 900 6 700	12 800 6 100	12 300 5 000	12 800 5 900	1510 3285
ZM J655 Alloy	2260 F. ½ hr., W.Q.	1500	15 000	5 000	4 900	4 800[c]		4 900	389
ZK J653 Alloy	2260 F. ½ hr., W.Q.	1500	15 000	7 500	6 800[c]	6 600[c]		7 000	225
ZW 6059 Alloy	2260 F. ½ hr., W.Q.; 1500 F. 50 hr., A.C.	1500 1500	15 000 15 000[b]	4 000 4 800	3 950 4 700	3 900 4 600	3 700 4 300	3 900 4 600	964 2136
ZS 6059 Alloy	2260 F. ½ hr., W.Q.	1500	15 000	8 400	6 700	6 100	. . .	6 700	569

[a] W.Q. = water quenched, F.C. = furnace cooled, A.C. = air cooled.
Same specimen as above test, reloaded.
[c] Extrapolated.

definitely. However, it appears to be in excellent condition after 20,000 hr. at 900 F.

The extensive information furnished to Project 16 by the U. S. Naval Engineering Experiment Station at Annapolis enables a number of illuminating comparisons to be made, especially as regards the difference in creep strength between values obtained in a constant stress test and the values given by the relaxation test.

Tables V and VI show the short-time tension test results both at room temperature and at elevated temperatures for the various materials contributed by the Engineering Experiment Station. Tables VII and VIII give the results of constant stress creep tests and 100 to 1000-hr. rupture tests. These figures enable comparisons to be made with residual stresses determined by means of relaxation tests as given in Tables IX and X.

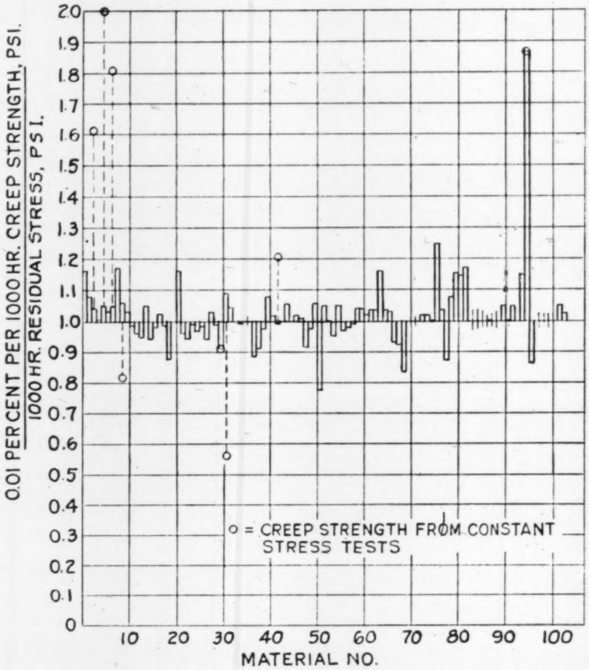


FIG. 10.—Correlation Between Nominal Creep Strength and 1000-hr. Residual Stress.

This chart shows the ratio between the creep strength having a rate of 0.01 per cent per 1000 hr. and the 1000-hr. residual stress. This chart includes all results from Tables I, II, and III regardless of composition or test temperature. The average of all these ratios for all the materials given in Table II, Nos. 9 to 72 inclusive, is 1.00. Where the creep strength has been determined from an independent series of constant stress tests on the same material, the ratio to the 1000-hr. residual stress is shown by the small circle. Such values range from 57 per cent to 200 per cent of the 1000-hr. residual stress.

Figures 8 and 9 illustrate the type of machine used in the Engineering Experiment Station for running these tests.

The results given in Tables VII and VIII enable a direct comparison between the creep strength as determined in the constant stress test and the creep strength determined in the relaxation test, the latter being the value which should be used in the formula in order to get an answer for the residual stress which has been determined in the relaxation test. As noted before, the creep strengths given in Part (*a*) of Tables I, II, and III are the figures determined in the relaxation test and suitable to be used in the formula to determine long-time bolt performance. Such figures may differ more or less from the results of constant stress tests. (One of the most important influences accounting for the difference between the two values is the fact that throughout a relaxation test the total elongation is strictly limited, whereas in the constant stress creep test, the total elongation is not limited but varies widely according to the applied stress, thus giving magnified differences from test to test due at least to a considerable degree to differences in strain hardening.)

An examination of Part (*a*) of Tables I, II, and III shows an interesting correlation between the creep strength for 0.01 per cent per 1000 hr. and the 1000-hr. residual stress. This ratio has been plotted for all listed references in Fig. 10. The small deviation from unity, either above or below, appears quite remarkable. An average of all these ratios for Part (*a*) of Table II comes out at 1.00, while for the most part the equality is better than 5 per cent.

Where constant stress creep strengths are available, the corresponding ratio is shown on Fig. 10 by a small circle. The spread from 2 to 1 above the line down to almost $\frac{1}{2}$ below shows that there is no easy correlation between the constant stress creep test results as ordinarily determined and the nominal creep strength which must be used in the formula to give the residual stresses in bolts.

Acknowledgment:

The author of this progress report desires to express his indebtedness to all the contributors[3] to this effort for their hearty cooperation in furnishing the original data which have made the report possible and to Mr. A. W. Wheeler in particular for his painstaking efforts in compiling the tables and diagrams.

DISCUSSION

Messrs. Vincent T. Malcolm and Sidney Low.[1]—It must be borne in mind, when the results of a simple relaxation test are studied, that the results, while quantitative in themselves, are qualitative when used to predict the behavior of a flanged joint at an elevated temperature.

An actual bolted, flanged joint consists of a combination of the following components: (1) studs, (2) nuts, (3) gasket, (4) thrust washers, and (5) flanges.

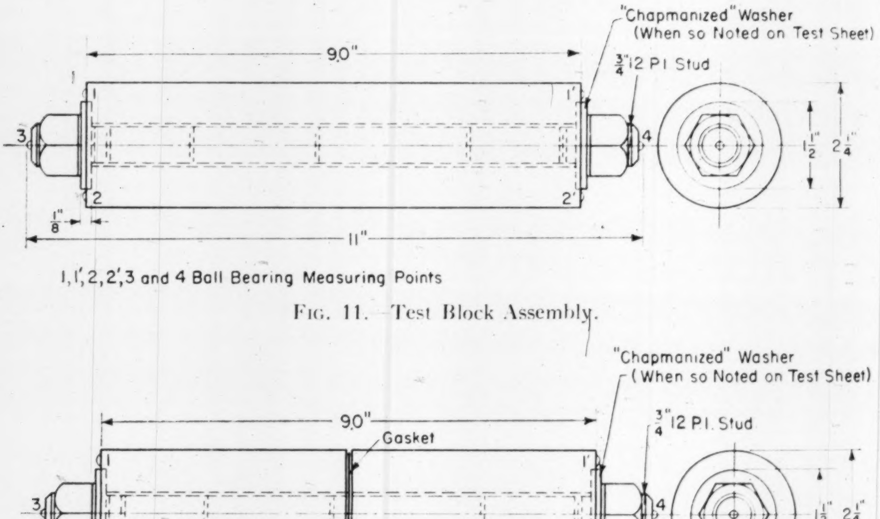

"Chapmanized" Washer
(When so Noted on Test Sheet)
9.0"
3/4" 12 P.I. Stud
1
1'
2
2'
3
4
1/8"
11"
1 1/2"
2 1/4"
1, 1', 2, 2', 3 and 4 Ball Bearing Measuring Points

Fig. 11.—Test Block Assembly.

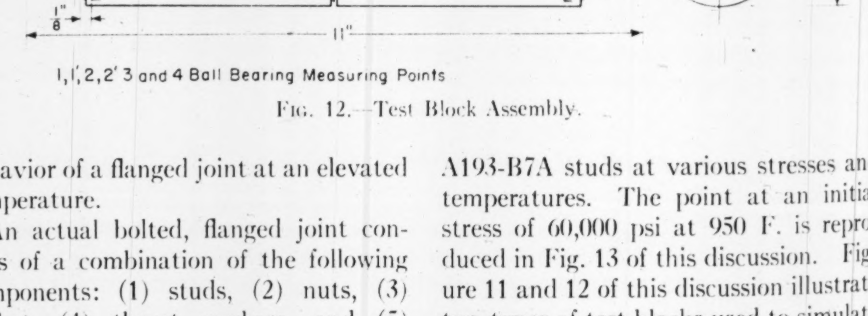

"Chapmanized" Washer
(When so Noted on Test Sheet)
9.0"
Gasket
3/4" 12 P.I. Stud
1
1'
2
2'
3
4
1/8"
11"
1 1/2"
2 1/4"
1, 1', 2, 2' 3 and 4 Ball Bearing Measuring Points

Fig. 12.—Test Block Assembly.

Mathematical methods are available to relate stud and flange behavior with respect to relaxation, but the other variables must be treated experimentally.

In Fig. 2 of the report data are presented on the relaxation behavior of A193-B7A studs at various stresses and temperatures. The point at an initial stress of 60,000 psi at 950 F. is reproduced in Fig. 13 of this discussion. Figure 11 and 12 of this discussion illustrate two types of test blocks used to simulate the relaxation of a joint. By relating simple stud relaxation tests and test block relaxation tests, we are able to predict flanged joint relaxation more accurately. Figure 13 illustrates this relationship. The effect of the various variables may be readily noted.

---

[1] Research Laboratory, The Chapman Valve Manufacturing Co., Indian Orchard, Mass.

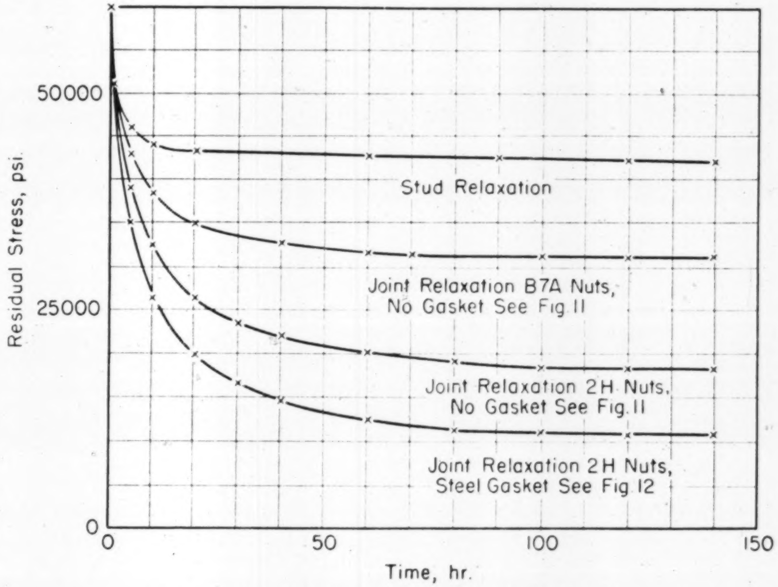


FIG. 13.—Joint Relaxation Test at 950 F.

Stud—A 193 B7A. *Nuts*—A 194 2H, Semi-finished, oil quenched, or A 193 B 7A bar stock nuts. *Sleeve*—A 193 B74 *Gasket*—SAE No. 1005 (copper plated). *Washers*—"chapmanized" carbon steel.

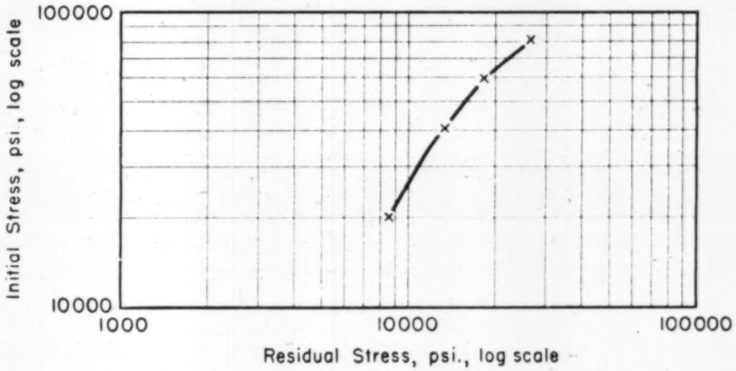


FIG. 14.—Joint Relaxation Test at 950 F.

Stud—A 193 B 7A. *Sleeve*—A 193 B 7A. *Nuts*—A 194 2H, oil quenched, semi-finished (see Fig. 11). Length of test, 150 hr.

Fortunately these block tests are a more severe test of relaxation behavior than is an actual flanged joint. The entire test block assembly is brought to test temperature while a very large temperature gradient exists across an actual flanged joint. Differentials as high as 500 F. have been reported for this gradient. Obviously, this gradient serves to reduce the relaxation of a joint by reducing the temperature of the various joint components.

It must also be borne in mind that another variable must be considered: the mechanical fit of the nut on the stud. Variations in both pitch diameters and lead of the nuts and studs affect the relaxation behavior of the joint. The nuts used in the tests reported herein are of commercial quality, Class 2 fit.

Figure 14 illustrates the effect of initial stress upon residual stress of a joint at a constant temperature.

MR. E. L. ROBINSON (*author's closure for the Committee*).—The comments contributed by Messrs. Malcolm and Low emphasize the importance of distinguishing between the behavior of a simple bolt in an unyielding flange and the more complicated designs frequently used. Where non-creeping elastic follow-up is provided, behavior may be predicted by estimating the elasticity factor as outlined in the first progress report for this project.[2] Where the design is too complex for rational analysis, the test procedure suggested by Messrs. Malcolm and Low may be used. This is, however, a matter of last resort. The object of Project 16 has been to point out the means whereby relaxation behavior may be as capable of prediction by rational processes as any behavior involving plastic action of the material.

---

[2] Ernest L. Robinson, "The Resistance to Relaxation of Materials at High Temperature," *Transactions*, Am. Soc. Mechanical Engrs., August, 1939, pp. 543–554.

APPENDIX II

STABILITY OF STEELS AS AFFECTED BY TEMPERATURE*[1]

(PROJECT NO. 29)

This report is the fourth of a series presenting laboratory test results on graphitization susceptibility near welded joints of steels developed for high-pressure high-temperature steam piping. Previous reports[2,3] have described materials, specimens, and test procedures employed in this investigation and contained data for various aging periods.

The steels being studied are as follows:

A.S.T.M. Designation	Grade	Nominal Composition, per cent			
		Carbon	Molybdenum	Chromium	Nickel
A 217	WC1	0.30 max.	0.40 to 0.60		
A 217	WC3	0.30 max.	0.40 to 0.60	0.50 to 0.80	
A 217	WC4	0.30 max.	0.40 to 0.60	0.40 to 0.70	0.75 to 1.05
A 217	WC6	0.20 max.	0.40 to 0.60	1.00 to 1.50	
A 157	C3A	0.30 max.	0.45 to 0.65	1.50 to 2.25	
A 280		0.10 to 0.20	0.45 to 0.65	0.50 to 0.70	

Detailed chemical analyses of individual heats have been presented in an earlier report.[3]

The castings program sponsored by the Manufacturers Standardization Society of the Valve and Fittings Industry has yielded information for WC1 and WC3 cast steels aged at 1025 F. for periods up to 15,000 hr. More highly alloyed materials, namely, WC4, WC6 and C3A cast steels, have been exposed at 1100 F. for as much as 6000 hr. Study of welding variables on the graphitization process in pipe conforming to A.S.T.M. Specifications A 280[4] was undertaken by the Pipe Fabrication Institute and results of 6000 hr. aging at 1100 F. have been made available.[5]

Carbon-molybdenum and chromium-molybdenum ($\frac{1}{2}$ per cent Cr) cast steels have been under test now for periods up to 25,000 hr. Results of examinations since the last report on these materials are shown in Table I, the evaluation being presented in the same manner as previously. A graphical representation Fig. 1, shows the rate of graphite formation from carbide for carbon-molybdenum steel treated with 2 lb. aluminum per ton (Group 1) to have decreased considerably after about 5000 hr. exposure at 1025 F. Data for other steels do not show a similar trend but in no case has graphitization progressed as far in these materials as in the Group 1 steels.

Trends indicated by earlier test results have been substantiated. Decrease in

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] This report was prepared by E. A. Sticha, Assistant Materials Research Engineer, Crane Co., Chicago, Ill., and submitted by J. J. Kanter, Chairman, Project No. 29, Materials Research Engineer, Crane Co., Chicago, Ill.

[2] J. J. Kanter, "Studies of Susceptibility of Casting Steels to Graphitization," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, pp. 581–587 (1946).

[3] J. J. Kanter and E. A. Sticha, "Studies of Susceptibility of Casting Steels to Graphitization," Appendix to Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 182 (1946).

[4] Tentative Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High-Temperatures (A 280-47 T), 1947 Supplement to Book of A.S.T.M. Standards, Part I-A, p. 306.

[5] J. J. Kanter and E. A. Sticha, "Stability of Steels as Affected by Temperature," Appendix to Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 207 (1947).

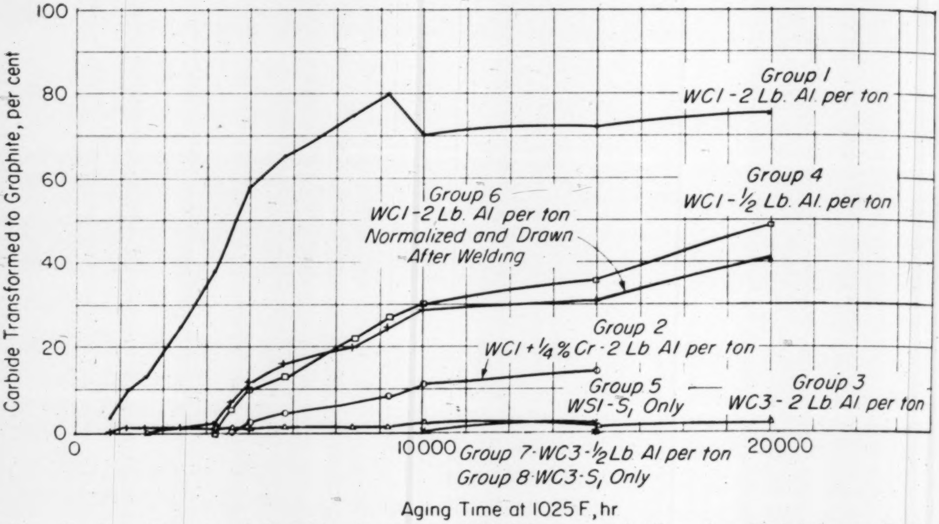


FIG. 1.—Development of Graphite in Carbon-Molybdenum and Chromium-Molybdenum Steels at 1025 F.

FIG. 2.—Graphite in "contact" zone of Silicon-Killed WC 1 Steel, Aged 15,000 hr. at 1025 F. (× 500).

FIG. 3.—Graphite in Base Metal of Aluminum-Killed WC 3 Steel, Aged 20,000 hr. at 1025 F. (× 500).

graphitization susceptibility is achieved by reduction of aluminum addition for deoxidation but complete elimination of this treatment does not guarantee freedom from possibility of graphite formation as shown in Fig. 2 by the presence of graphite in silicon-killed WC1 steel after 15,000 hr. aging at 1025 F. This experience confirms similar findings of other investigators.[6] As before, Group 6 steels, normalized and drawn after welding, show about the same graphitization tendencies as Group 4 steels, WC1 analysis treated with ½ lb. aluminum per ton.

Effectiveness of chromium additions in retarding formation of graphite in carbon-molybdenum steels has been further demonstrated. There has been little, if any increase in the percentage of graphite developed between 10,000 and 20,000 hr. aging at 1025 F. in steel containing ½ per cent chromium and deoxidized with 2 lb. aluminum per ton. However, significant amounts of graphite were observed in metal of this composition which had not been subjected to the welding operation or subsequent heat treatments (Fig. 3). Thus, additon of ½ per cent chromium cannot in itself be considered sufficient to preclude formation of graphite at 1025 F. in steel deoxidized with 2 lb. aluminum per ton.

This portion of the casting program has fulfilled its purpose of evaluating certain deoxidation practices, heat treatments after welding, and efficacy of chromium additions with respect to graphitization susceptibility and might well be concluded in the near future. Continued exposure of some of the more resistant steels might be advisable in order to establish better the trends shown in Fig. 1.

TABLE I.—ESTIMATED PORTION IN PER CENT OF CARBIDE TRANSFORMED TO GRAPHITE FOR WC1 AND WC3 CAST STEELS AGED AT 1025 F.

	Mark	Aged 10 000 hr.	Aged 15 000 hr.	Aged 20 000 hr.	Aged 25 000 hr.
Group 1—WC1, deoxidized with 2 lb. Al per ton.	1 B			50	
	2 B				90
	4 B			75	
	5 B				75
	7 B	75	75	75	
	7 BI	90	90	90	
	Average	70	72	75	
Group 2—WC1 plus ¼ per cent Cr, deoxidized with 2 lb. Al per ton.	6 B				25
	7 B′		0		
	7 *		0		
	7 –		25		
	7 X		25		
	Average		14		
Group 3—WC3, deoxidized with 2 lb. Al per ton.	1 C			0	
	2 C				0
	4 C			0	
	5 C				0
	7 C		0	0	
	7 CI		5	10	
	7 C′		0		
	Average		1	2	
Group 4—WC1, deoxidized with ½ lb. Al per ton.	1 A			5	
	2 A				75
	4 A			50	
	5 A				25
	7 A	25	25	75	
	7 AI	50	75	75	
	Average	30	36	49	
Group 5—WC1, deoxidized with Si only.	3 D	0	0		
	4 D	0	0		
	5 D	0	10		
	6 DIs				0
	7 DI	0	0	0	
	Average	0	2		
Group 6—WC1, deoxidized with 2 lb. Al per ton. Normalized and drawn after welding.	1 BH			25	
	2 BH				75
	4 BH			50	
	5 BH				75
	6 BH				25
	7 BH		10	25	
	7 BIH		50	50	
	Average		31	41	
Group 7—WC3, deoxidized with ½ lb. Al per ton.	2 E	0	0		
	4 E	0	0		
	5 E				0
	Average	0	0		
Group 8—WC3, deoxidized with Si only.	4 G			0	
	5 G	0	0		
	Average	0	0		

---

[6] A. J. Smith, J. B. Urban and J. W. Bolton, "Graphitization in Some Cast Steels," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, pp. 609–620 (1946).

The casting program also included study of more highly alloyed steels, all deoxidized with 2 lb. aluminum per ton. These materials are being held at 1100 F. and have shown no graphite thus far. Under test are six specimens of WC4 analysis, four of which have been aged for 10,000 hr.; ten of WC6 analysis, two aged for 15,000 hr. and seven for 10,000 hr.; five of C3A analysis, two each aged for 10,000 and 15,000 hr.; eight of WC3 analysis, one aged for 15,000 hr. and four for 10,000 hr. In general, these samples show some agglomeration of carbides, there being a slight tendency for concentration in grain boundaries.

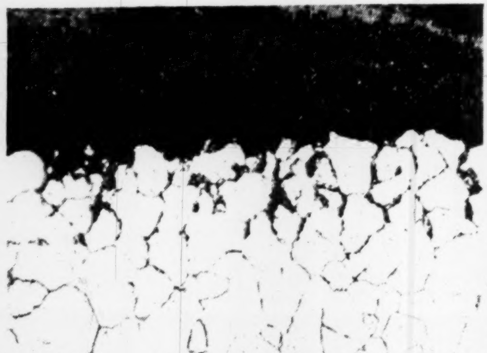


FIG. 4.—Intergranular Penetration of Oxygen under Scale Between A 280 Pipe and Backing Ring, Aged 13,000 hr. at 1100 F. (X 500).

Some of these steels are being exposed at 1025 F. also, and no graphite has developed at this temperature either. Of three WC4 steels, two have been aged for 15,000 hr., of two WC6 steels one has been aged for 15,000 hr., and of six C3A steels five have been aged for 15,000 hr. This phase of the casting investigation requires much more prolonged exposures before these materials can be evaluated satisfactorily.

Aging of weld specimens of Specifications A 280 pipe, has been under way for 13,000 hr. at 1100 F. and no graphite has been observed. This result is in agreement with that for cast steel of similar composition and deoxidation treatment, Group 7 as of Table I. Carbides in the heat-affected zone have agglomerated somewhat and there is evidence of some precipitation in ferritic areas which, however, seems to be no greater than at the previous examinations.

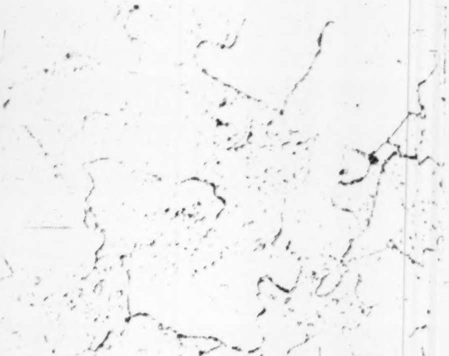


FIG. 5.—Markings in Ferrite along Inner Pipe Wall near Weld, Aged 13,000 hr. at 1100 F. (X 500).

Some interesting developments have been observed in the structure of these samples. Figure 4 shows intergranular penetration of oxygen under the heavy scale formed between the pipe and backing ring. Since exposure is at 1100 F. it is unlikely that this attack is of consequence in the anticipated scope of application for this material, but it serves as a warning to avoid operation at too high a temperature level. A second feature noted in some of the specimens is markings similar to Neumann bands or nitride needles in ferrite grains along the inner pipe wall and a short distance from the weld (Fig. 5). These markings were not noted in the original welded structure but were observed after 1500 hr. aging and thereafter. Their appearance has not changed appreciably in this time hence there seems to be very little possibility of trouble from this source. Some importance was attached to the bands because of the findings of Emerson and Morrow with respect to the Springdale pipe.[7] Further aging is indicated for this material.

At a recent meeting of Project No. 29 it was decided that investigation into the fundamental nature of the graphitization process was needed and arrangements for this study are proceeding with Battelle Memorial Institute. The work is to embrace examinations by chemical, spectrographic, and X-ray diffraction methods of solutions and residues obtained by the electrolytic extraction of carbides from two carbon steels, one of known high susceptibility to graphite formation by virtue of a large aluminum addition and the other of low susceptibility. While carbon-molybdenum steel would perhaps have been preferred as the material for study, experimental difficulties and complications in interpretation due to the presence of molybdenum indicated that this choice would be unwise.

Our knowledge of factors which promote development of graphite in low-alloy steels at high temperatures has progressed to the point where practical means of preventing graphite formation are well known and further studies should be of a more fundamental character to reveal the mechanism of the process.

Castings and investigational services for the Manufacturers Standardization Society of the Valve and Fittings Industry program were supplied by Chapman Valve Mfg. Co., Crane Co., Edward Valves, Inc., Lunkenheimer Co., Wm. Powell Co., Reading-Pratt & Cady Division of the American Chain and Cable Co., and Walworth Co. Cooperating in the Pipe Fabrication Institute study are American Locomotive Co., Crane Co., Grinnell Co., M. W. Kellogg Co. and Pittsburgh Piping and Equipment Company.

---

[7] R. W. Emerson and Mathew Morrow, "Further Observation of Graphitization in Aluminum-Killed Carbon-Molybdenum Steel Steam Piping," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, pp. 597–607 (1946).

REPORT OF COMMITTEE C-1* ON CEMENT

Committee C-1 on Cement held two meetings during the past year: one in November, 1947, at Chicago, Ill.; the other in February, 1948, at Denver, Colo. The attendance of members at the Chicago meeting was one of the largest in the history of the committee. An additional feature was a visit to Naperville, Ill. to inspect the long-time concrete test specimens of the Portland Cement Association. Transportation was kindly arranged by that Association. Naperville is one of the widely different exposure stations used in that Association's current extensive study of portland cements with respect to durability over a period of years.

The committee records with sorrow the death of Joseph C. Pearson who had been an active member of the committee for almost thirty years.

The subcommittees have been very active throughout the year, as evidenced by cooperative studies and subcommittee reports.

The Sponsoring Committee on Portland Cement (R. R. Litehiser, chairman) has considered numerous questions relating to the specifications for portland cement, particularly the matter of limits for alkali content of cements that are to be used with reactive aggregates. Recommendations made by the sponsoring committee have resulted in proposals, presented later in this report, affecting the Specifications for Portland Cement (C 150) and for Air-Entraining Portland Cement (C 175). Questions relating to the chemical requirements for type V cement are also being studied.

The Sponsoring Committee on Masonry Cement (H. D. Baylor, chairman) has continued its studies toward a durability specification for masonry cement. A new series of tests is being undertaken, using a 1:3 mortar test specimen that is larger in cross-section than the specimens that were employed in the preceding series and had been found somewhat fragile for the purpose. In the earlier series, seven laboratories had participated in the tests of eight cements. In line with the sponsoring committee's proposals, the committee is now presenting, elsewhere in this report, recommendations affecting the Specifications for Masonry Cement (C 91 – 40 and C 91 – 44 T). The sponsoring committee is also actively studying questions relating to the Specifications for Natural Cement (C 10) and is collecting data to assist in revising that standard.

The Sponsoring Committee on Blended Cements (L. R. Forbrich, chairman) is continuing its studies of tests for pozzolanic activity. The sponsoring committee also presented a revision, as appended hereto,[1] of the Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205) incorporating therein a provision for the use of air-entraining agents now allowed under Specifications C 175 so as to afford an air-entraining portland blast-furnace slag cement.

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) completed its cooperative study of methods for determining titanium oxide in portland cement. Eight laboratories assisted in this work. The subcommittee is recommending three new methods for this determination, as appended hereto,[2] for inclusion in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 46 T). The working committee is continuing its studies of chemical methods that are of interest to cement users.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] These revised specifications were accepted by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

The Working Committee on Volume Change and Soundness of Portland Cement (H. F. Gonnerman, chairman) reviewed the extensive literature of recent years relating to the chemical reactions of aggregates in concrete, and furnished Committee C-1 members with an eight-page summary of the various methods of test for the reactivity of aggregates in mortar and concrete. The working committee is continuing its efforts to establish a test procedure for determining whether or not deleterious reactions are likely to occur with a given combination of cement and aggregate.

The Working Committee on Sulfate Resistance (D. Wolochow, chairman) studied available recent data and made plans for new cooperative tests. Three laboratories are participating in a pilot study of 1:5 mortar bars (normal consistency), and 1:4 plastic mortar bars (wet and dry) made in accordance with method C 109. Thereafter, twelve laboratories plan to participate in a larger series of tests.

The Working Committee on Time of Setting (E. E. Berger, chairman) completed the study of the results of two series of cooperative tests, in which seven laboratories had tested four cements, each cement being of a different type. Based on the results of those tests, the subcommittee presented a Proposed Method of Test for Setting Time of Hydraulic Cement in Mortar wherein a modified Vicat apparatus is used for measuring consistency and determining setting time of hydraulic cement in mortar. The proposed method, published as information only at this time, is appended to this report.[3]

The Working Committee on Heat of Hydration (G. J. Durant, chairman) has been engaged in studying questions relating to the heat of hydration of portland cement at early ages.

The Working Committee on Bleeding, Plasticity, and Workability (H. L. Kennedy, chairman) studied a suggested new method for preparing tensile strength briquets. The subcommittee also initiated an extensive study of questions relating to the flow table. A tentative flow table design was circulated among those interested in order to promote discussion and suggestions that may assist in preparing a satisfactory specification for that apparatus. The subcommittee outlined a tentative program directed toward establishing a test for the bleeding of portland-cement pastes and mortars.

The Working Committee on Fineness (R. L. Blaine, chairman) has been considering details related to the turbidimeter fineness test, particularly the merits of different dispersing agents, and the possibility of determining the size and percentage of material finer than 7.5 microns. Tentative plans for cooperative work by the committee members were made.

The Working Committee on Strength (G. L. Lindsay, chairman) made a study of certain test data on the relationship between the tensile, compressive, and flexural strengths of mortar and the flexural strength of concrete. Upon the completion of that study, the working committee planned and began a cooperative investigation for the purpose of developing information as to the relative merit of tensile, compressive, and flexural strengths of mortar specimens in predicting the flexural and compressive strengths of concrete. Fifteen laboratories, nine of which are state highway departments, participated in this investigation. Four cements (types I, IA, II, and III) were used in the work. The work is well under way, some of the laboratories having about completed their work.

---

[2] These methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

[3] See p. 250.

The Working Committee on Additions (H. J. Gilkey, chairman) completed the study of the data resulting from the tests made by the Cement Reference Laboratory under the subcommittee's directions, on a material that had been submitted to Committee C - 1 as an addition to cement under Specifications C 150. Based on the subcommittee's recommendations, Committee C - 1 has declared that the material 109 - B (2 methyl 2–4 pentane diol), tested at the request of the Master Builders Co., is acceptable for inclusion in Specifications C 150 as a nonharmful addition when added in an amount not exceeding 0.03 per cent by weight of the cement, except that in type III cement a maximum of 0.05 per cent may be used. In presenting the related recommendations, the working committee invited attention to the fact that material 109 - B, when added within the limits above mentioned, entrains air to a slight degree, but not sufficient to bring the air-entrainment within the limits recommended by Specifications C 175. The subcommittee also presented to Committee C - 1 a proposed definition of "addition," which is included later in this report.

The Working Committee on Coordination of Methods of Test (J. R. Dwyer, chairman) assisted in arranging for the use of an improved shipping container for standard Ottawa sand, designed to afford more protection against contamination during shipment and storage. The subcommittee is also maintaining contact with the developments in the move to secure a generally accepted standard for laboratory atmosphere conditions.

The Working Committee on SO_3 Content (H. S. Meissner, chairman) is engaged in studies in which seven laboratories are participating in the tests of eight cements, including four types. Each cement has been ground with seven different SO_3 contents. Results have been obtained from miscellaneous tests of the cements, including heat of hydration, autoclave expansion, and chemical analysis. Three of the laboratories have also contributed data on the use of the five proposed criteria for determining the optimum amount of gypsum for a given cement. The very close agreement secured among those three laboratories led the subcommittee to believe that an early start could be made on the second part of the investigation, concerning the effect of the SO_3 content on concrete durability.

The Working Committee on Methods of Test for Air-Entrainment (H. K. Cook, chairman) studied suggestions relating to the design of the Burmister flow trough that is used in determining the water requirements for the standard mortar in Tentative Method C 185. The subcommittee also outlined a series of cooperative tests that are to include: (*1*) The present Tentative Method C 185, (*2*) Current Federal Government test in which the flow table is employed, (*3*) The constant water content method employed by the Dewey & Almy Chemical Co. Ten laboratories are expected to participate in the tests of nine cements that will include types IA, IIA, and IIIA.

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) continued to supervise and assist in the work of the Cement Reference Laboratory at the National Bureau of Standards. That laboratory, sponsored by Committee C - 1, is a joint project of the Government and the A.S.T.M., and is supported by funds received through the National Bureau of Standards, the A.S.T.M., and the Public Roads Administration. The work of the laboratory during the past year is briefly outlined as follows.

Inspection service among cement testing laboratories was continued, but at a rate slower than usual because of reduced force. Fifty-five laboratories were inspected, mostly located in the east and the south. Fifty compression testing machines were verified. Efforts to secure additional help have been steadily continued, and it is hoped that the personnel may be increased soon.

The light trucks furnished by the National Bureau of Standards have helped greatly. More equipment can be carried than in the inspectors' own cars. Gage-testing equipment has been added so that the pressure gages on the autoclaves can be checked. Arrangements were recently made to secure additional proving rings so that compression machines in the cement laboratories can be verified over a larger range in load.

A study was directed toward developing a mixture that could be used for calibrating the 10-in. flow table. Related work had been done by others in recent years. Prompted by their work, and guided by assistance from other laboratories in the Bureau, a number of materials were investigated. A mixture was finally selected for use in some extended trials. It was found possible to reproduce satisfactorily the very finely ground material. A trial lot of about 25 samples was distributed to certain interested laboratories, including the laboratories that were participating in the cooperative strength tests sponsored by the Working Committee on Strength. Most of those samples will be returned to the Reference Laboratory for retest at an early date. Some of the earlier samples, used over and over many times, have retained their flow characteristics practically unchanged for long periods. The inspectors have been using the mixture in their field work to aid in checking and adjusting flow tables, and the information that is being collected should be helpful in preparing specifications for the flow table.

Cooperating with the Working Committee on Additions, the laboratory prepared a condensed report of the tests that the laboratory had made during the past two years on cements in which the material 109-B had been used as an addition.

The head of the laboratory attended two meetings of Committee C - 1 and numerous subcommittee meetings. He also attended conferences relating to plans for the laboratory's work. The laboratory was represented at various subcommittee meetings, Federal and ASTM, where matters relating to laboratory equipment, methods, and conditions were being considered. The laboratory cooperated with a Government agency in assembling data to assist in planning for a Government laboratory project.

Miscellaneous work has included the testing of cement laboratory equipment that was received at the Bureau for Reference Laboratory inspection.

In line with similar action in other committees of the Society, Committee C - 1 has formed an Editorial Subcommittee, consisting of W. J. McCoy (chairman), L. R. Forbrich, and F. O. Slate.

NEW TENTATIVE

Tentative Methods for Determining Titanium Oxide in Portland Cement.—There has been increasing interest regarding some of the minor constituents in portland cement, and the committee now recommends the acceptance as tentative of three methods for determination of TiO_2. These methods appear in Appendix I[2] to this report, and are offered for incorporation as parts of the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 46 T). Extensive cooperative study has not furnished conclusive evidence as to which of the three methods is best, but during such time as they may be used as tentative sufficient data may become available to enable the selection of a referee method.

REVISION OF TENTATIVES

Tentative Specifications for Air-Entraining Portland Cement (**C 175 - 47 T**).[4] —In order to provide requirements covering an air-entraining "type III" cement, the committee recommends that these specifications be revised by the addition of the following requirements for a cement to be designated as "type IIIA":

Table I.—Insert for the new type IIIA cement the same chemical requirements as are now shown for type III cement in Table I of the Standard Specifications for Portland Cement (C 150 - 47).[4]

Table II.—Insert for the new type IIIA cement the same physical requirements for soundness and time of setting as are now shown for type III cement in Table II of Standard Specifications C 150; also add the following requirements:

Air content of mortar (prepared and tested in accordance with Method C 185), per cent by volume....	18 ± 3
Compressive strength:	
1 day in moist air...............	1100 psi.
1 day in moist air, 2 days in water	2200 psi.

Make the other necessary changes in the text to take care of the addition of this new type IIIA cement.

In presenting the above recommendation, the committee invites attention to the fact that the proposed compressive strength requirements are the same as the corresponding requirements in the present Federal Specification for type IIIA cement.

Tentative Specifications for Portland Blast-Furnace Slag Cement (**C 205 - 47 T**).[4]—The committee recommends that these specifications be revised as appended hereto.[1] These revisions are recommended in order to provide requirements covering an air-entraining portland blast-furnace slag cement.

TENTATIVE REVISION OF STANDARD

Standard Specifications for Portland Cement (**C 150 - 47**).—The committee recommends the following tentative revision in the strength requirements of these specifications:

Table II.—For type V cement, change the requirements for compressive strength at 7 days from "1000 psi." to read "1500 psi." and at 28 days from "2200 psi." to read "3000 psi."

For type V cement, change the requirements for tensile strength at 7 days from "175 psi." to read "250 psi." and at 28 days from "300 psi." to read "325 psi."

These revisions make the requirements for compressive and tensile strength at 7 and 28 days for type V cement the same as the respective strengths now required for type II cement in the same table.

As explained in another part of this report, the committee had reviewed the results of tests made by the Cement Reference Laboratory on material 109-B (2 methyl 2-4 pentane diol), and had declared that material acceptable for inclusion in Standard Specifications C 150 as a non-harmful addition when added in an amount not exceeding 0.03 per cent by weight of the cement, except that in type III cement a maximum of 0.05 per cent may be used. Material 109-B, when added within the limits above mentioned, entrains air to a slight degree, but not sufficient to bring the air-entrainment within the limits recommended by Specifications C 175. In keeping with the procedure described in Section 3 of C 150, the following information as to material 109-B is to be included as a revision of footnote 3 in Section 3 of Specifications C 150:

---

[4] 1947 Supplement to Book of A.S.T.M. Standards, Part II.

> [3] The committee has declared as not harmful the inclusion of the following materials:
>
> (*a*) The material known commercially as TDA (composed of triethanolamine and highly purified soluble calcium salts of modified lignin sulfonic acids), manufactured by the Dewey & Almy Chemical Co., when added in an amount not exceeding 0.043 per cent by weight of the cement, except that in type III cement a maximum of 0.08 per cent by weight may be used.
>
> (*b*) The material known commercially as "109-B" (composed essentially of 2 methyl 2-4 pentane diol), marketed by the Master Builders Co., when added in an amount not exceeding 0.03 per cent by weight of the cement, except that in type III cement a maximum of 0.05 per cent by weight may be used.

ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Masonry Cement (**C 91 – 44 T**).[5]—These tentative specifications have remained unchanged for over three years, during which time study has been given to related questions. The committee now recommends that these tentative specifications be approved for reference to letter ballot of the Society for adoption as standard to replace the present Standard Specifications C 91 – 40. The committee accordingly recommends that present Standard Specifications C 91 – 40 be withdrawn upon the adoption of C 91 – 44 T as standard.

DEFINITION

Study was made of the questions that had arisen regarding the meaning of "additions" in discussions relating to cement. The committee now recommends that the following definition be accepted by the Society for publication as a tentative definition:[6]

> *Addition.*—In the manufacture of portland cement, any material other than water and/or untreated calcium sulfate, which is interground with the clinker.

TENTATIVE METHOD CONTINUED

Pending the completion of current study of the air-permeability fineness test, it is recommended that Tentative Method C 204 – 46 T be retained as tentative without change.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

The election of officers for the ensuing term of two years resulted in the re-election of the present incumbments.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members, of whom 73½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

G. E. WARREN,
Secretary.

---

[5] 1946 Book of A.S.T.M. Standards, Part II.

[6] This definition was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards a revision of Tentative Specifications for Air-Entraining Portland Cement (C 175 – 47 T). This recommendation was accepted by the Standards Committee on September 29, 1948, and the revised tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

APPENDIX

PROPOSED METHOD OF TEST FOR SETTING TIME OF HYDRAULIC CEMENT IN MORTAR[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended for determining the setting time of hydraulic cement in a one to one mortar.

Apparatus

2. (*a*) *Scales.*—Scales having a capacity of 1000 g. or more shall be used. The permissible variation at 1000 g. shall be plus or minus 1 g.

(*b*) *Weights.*—The permissible variations on weights used in weighing the cement and sand shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
500	0.35
300	0.30
250	0.25
200	0.20

(*c*) *Glass Graduate.*—A glass graduate made to deliver 250 ml. at 20 C. shall be used. The graduate shall have graduations for each 2 ml. and the permissible variation of the graduate shall be plus or minus 2 ml.

(*d*) *Tamper.*—The tamper shall be made of nonabsorptive, nonabrasive material, and shall have a cross-section of $\frac{1}{2}$ by 1 in. and a convenient length of 5 or 6 in.

(*e*) *Vicat Apparatus Modified for Consistency Determination.*—The penetration apparatus used for measuring consistency shall be similar to the Vicat apparatus described in Section 2(*d*) of the Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191),[2] except that it shall be modified as follows: The total weight of the plunger shall be 400 g. One end shall be similar to that used for measuring consistency except that it shall be $\frac{11}{16}$ in. in diameter. This modification may be accomplished by substituting for the 1-mm. needle an $\frac{11}{16}$-in. brass rod, $2\frac{1}{8}$ in. in length and provided with a stud at one end for fastening to the plunger. This rod will weigh approximately 100 g. so that with a small adjustment the assembled plunger can be made to weigh 400 g. The mold for containing the mortar shall be a smooth cylindrical brass ring 3 in. in inside diameter by 1 $\frac{11}{16}$ in. in depth.

(*f*) *Vicat Apparatus Modified for Setting Time Test.*—The regular Vicat apparatus described in Section 2(*d*) of A.S.T.M. Methods C 191 shall be used except that a 2-mm. needle shall be substituted for the 1-mm. needle and the total weight of the plunger shall be readjusted to 300 g. The needle shall be made from a stiff wire such as piano wire. The mold shall consist of a cylindrical brass ring 3 in. in inside diameter by $1\frac{11}{16}$ in. in depth cemented to a glass plate, or a brass cup of the same dimensions may be used.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement. Published as information, January, 1948.

[2] 1946 Book of A.S.T.M. Standards, Part II.

(*g*) *Mixing Bowl.*—A stainless steel mixing bowl shall be used. The bowl shall have a capacity of 6 qt. and be similar in shape and dimensions to the bowl shown in Fig. 2 of the Tentative Method of Test for Air Content of Air-Entraining Portland-Cement Mortar (A.S.T.M. Designation: C 185).[3]

Temperature and Humidity

3. The temperature of the room and the temperature of the sand and cement used shall be 72 ± 4 F. The temperature of the mixing water and moist cabinet shall be 70 ± 2 F. The relative humidity of the moist cabinet shall be greater than 90 per cent.

Procedure

4. (*a*) *Batch.*—This mix shall consist of 750 g. of cement and 750 g. of natural silica sand from Ottawa, Ill., graded as specified in Sections 4 and 5 of the Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (A.S.T.M. Designation: C 109).[3] Approximately 30 to 33 per cent water by weight of the cement will be required, depending upon the cement tested. The use of distilled water is preferable, and distilled water shall be used in referee tests or cooperative tests.

(*b*) *Mixing of Mortar.*—The mixing shall be done in a bowl (Section 2(*g*)) by continuous stirring, squeezing, and kneading with one hand which shall be protected by a snug fitting rubber glove. The material for the batch shall be introduced in the following manner:

(*1*) Place the water in the bowl which has just been wiped with a damp cloth,

(*2*) Add the cement to the water and mix for 30 sec.,

(*3*) Add approximately one half of the sand and mix for 1 min.,

(*4*) Add the remainder of the sand and mix for 1 min.,

(*5*) Let the mortar stand for $1\frac{1}{2}$ min. covered by a damp cloth, keeping the rubber glove covered also, and

(*6*) Mix for $1\frac{1}{2}$ min.

(*c*) *Method of Filling Mold for Consistency Measurement.*—The mold (see Section 2(*e*)), sealed to a glass plate as described in Section 7 of Methods C 109, shall be filled in two layers as follows: Mortar shall be placed in the mold as level as possible without disturbance and in sufficient amount to fill the mold about half full. This shall be puddled 15 times with the tamper (Section 2(*d*)), making a complete circle around the outside of the mold with the 1-in. side of the tamper parallel to the radius of the ring, and then puddled by 3 more strokes across the center of the ring, making 18 strokes in all. The mold shall be filled to overflowing and the same puddling procedure repeated. The mortar then shall be cut off level in two operations as follows: First, with the trowel held at about a 30-deg. angle, start at one side of the mold $\frac{1}{2}$ in. above the mold and finish the stroke by cutting the mortar off the top of the mold at the opposite side. Second, by starting from the opposite direction as when using the trowel, take a straightedge held vertically and with a sawing motion cut the mortar flush with the top of the mold.

(*d*) *Penetration Test for Consistency.*—The mortar confined in the mold shall be placed in position. The plunger of the modified Vicat apparatus (Section 2(*e*)) shall be placed in contact with the surface of the mortar at the center of the ring and the set screw tightened. The movable indicator shall be placed at zero. The plunger then shall be released quickly by releasing the set screw and the penetration determined. The mortar shall have the desired consistency when the plunger settles to a point 20 ± 5 mm. below the surface 30 sec. after being released. The penetration test shall be made 35 to 50 sec. after starting to fill the mold. The bowl containing the remainder of the mortar shall be covered with a damp cloth while the consistency test is being made.

---

[3] 1947 Supplement to Book of A.S.T.M. Standards, Part II

(*e*) *Setting Time Test.*—Immediately after it has been found that a mortar of proper consistency has been obtained, the mortar remaining in the bowl shall be mixed an additional 30 sec. and the 3-in. mold shall be filled in exactly the same manner as specified for the consistency determination (Paragraph (*c*)). As soon as the mold is filled it shall be placed in the moist cabinet where it shall remain except when penetration tests are being made. In the penetration test for time of set, the modified Vicat apparatus specified in Section 2(*f*) shall be used and the same technique shall be used as given in Paragraph (*d*) for the consistency determination. The penetration shall be checked every 30 min. until the 2-mm. needle fails to penetrate to the bottom of the mortar, after which the penetration shall be checked every 10 min. for type III cement and every 15 min. for the other types of cement until a penetration of 10 mm. or less is obtained. No penetration test shall be made closer than $\frac{1}{4}$ in. from the former penetration position. Record the results of all penetration tests and by interpolation determine the time when a penetration of exactly 10 mm. would be obtained. This is the setting time.

(*f*) *Precautions.—All of the apparatus shall be free from vibration during the penetration tests.* The plunger of the Vicat apparatus shall not be oiled at any time, but the plunger and guide bearings shall be kept clean at all times, and at the beginning of each day's test a film of dry graphite shall be rubbed on the plunger.

REPORT OF COMMITTEE C-2*
ON
MAGNESIUM OXYCHLORIDE CEMENTS

Committee C-2 on Magnesium Oxychloride Cements held its organization meeting in Washington, D. C., on May 22, 1947. A second meeting was held on March 1, 1948, also in Washington, D. C.

The scope, as now adopted by Committee C-2 and approved by the Board of Directors of the Society, is as follows:

> *Scope.*—The stimulation of research and the formulation of specifications, methods of testing, and definitions relating to magnesium oxychloride cements.

At present, the committee consists of 16 members, of whom 8 are classified as producers, 7 as consumers, and 1 as a general interest member.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. S. Wells.
Vice-Chairman, Dean S. Hubbell.
Secretary, G. J. Fink.

Subcommittee II on Methods of Test, under the chairmanship of W. J. Riley, has prepared nine methods of test which have been submitted to Committee E-1 on Methods of Testing for review. These methods will be presented to the society through the Administrative Committee on Standards for publication as tentative. In addition, this subcommittee has developed seven other methods which are being circulated for letter ballot of the committee and which will then be submitted to the Standards Committee for acceptance as tentative. It has held three meetings and has reviewed preliminary drafts of several other methods. It is now actively engaged in the development of methods of test for soundness for the determination and prediction of volume changes and other characteristics of oxychloride flooring by means of which its reasonable life and service would be assured. This subcommittee also sponsored a paper entitled "New Type Weighted Needles for Determining the Setting Times of Magnesium Oxychloride Cements,"[1] by Harry E. Chisholm, which was presented at the Annual Meeting of the Society in June, 1948.

The Working Subcommittee on specifications has deferred the holding of a meeting, pending action of the Subcommittee on Methods of Test.

A special subcommittee expects to present by-laws for the consideration of Committee C-2.

This report has been submitted to letter ballot of the committee, which consists of 16 members; 16 members returned their ballots, of whom 13 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. S. Wells,
Chairman.

G. J. Fink,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] ASTM Bulletin, No. 154, October, 1948, p. 37 (TP 195).

REPORT OF COMMITTEE C-4* ON CLAY PIPE

Committee C-4 on Clay Pipe held one meeting during the year, in Atlantic City, N. J., on June 17, 1947.

During the year the committee has continued its growth, reaching a present total active membership of 30, of whom 13 are classified as producers, 10 as consumers, and 7 as general interest members.

The present officers of the committee have been reelected for the ensuring term of two years.

Subcommittee II on Specifications, under the chairmanship of D. G. Miller, was given the task of reviewing the following standard and tentatives which have stood for more than two years without revision, with instructions to report any recommended revisions at the next meeting of Committee C-4:

Standard Recommended Practice for:

Laying Sewer Pipe (C 12–19),

Tentative Specifications for:

Standard Strength Clay Sewer Pipe (C 13 - 44 T), and

Extra Strength Clay Pipe (C 200 - 44 T).

Recommendation Accepted by the Administrative Committee on Standards

Subsequent to the 1947 Annual Meeting, Committee C-4 presented to the Society through the Administrative Commitee on Standards, the proposed Tentative Specifications for Standard Strength Perforated Clay Pipe. The new tentative was accepted by the Standards Committee on September 4, 1947, and appears in the 1947 Supplement to the Book of A.S.T.M. Standards, Part II under the designation C 211 - 47 T.

This report has been submitted to letter ballot of the committee, which consists of 30 members; 28 members returned their ballots, of whom 27 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. C. Riedel,
Chairman.

Robert G. Scott,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

REPORT OF COMMITTEE C-7*

ON

LIME

Committee C-7 on Lime held two meetings during the past year: the first was at Atlantic City, N. J. on June 18, 1947, during the course of the Annual Meeting, and the second at Chicago, Ill., on November 3, 1947.

The committee now consists of 47 members of whom 23 are classified as producers, 6 as consumers and 18 as general interest members.

The Board of Directors of the Society has approved the following revised scope of the committee:

Scope.—The formulation of specifications, methods of test, and definitions for lime and lime products, and for limestone as it may be used in the chemical, agricultural, and process industries.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. C. Voss.
Vice-Chairman, E. E. Eakin.
Vice-Chairman, L. K. Herndon.
Secretary, R. S. Boynton.

Committee C-7 has taken a poll of all tentatives and standards under its jurisdiction to establish the need for their review or revision. Action has been taken to refer the following four tentatives to letter ballot of the committee for adoption as standard in 1949 and for subsequent publication in the 1949 Book of A.S.T.M. Standards:

Tentative Specifications for:

Normal Finishing Hydrated Lime (C 6 – 46 T),
Special Finishing Hydrated Lime (C 206 – 46 T),
Hydrated Lime for Masonry Purposes (C 207 – 46 T), and

Tentative Methods of:

Physical Testing of Quicklime and Hydrated Lime (C 110 – 45 T).

The following standards were considered by the committee to be in accord with present practice:

C 25 – 47
C 35 – 39
C 45 – 25
C 46 – 27
C 47 – 27
C 48 – 24
C 49 – 42
C 50 – 27
C 53 – 39
C 141 – 42

This report has been submitted to letter ballot of the committee, which consists of 47 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WALTER C. VOSS,
Chairman.

ROBERT S. BOYNTON,
Secretary.

---

*Presented at the Fifty-first **Annual Meeting of the** Society, June 21–25, 1948.

REPORT OF COMMITTEE C-8* ON REFRACTORIES

Committee C-8 on Refractories had two meetings during the year, the 72nd at Granville, Ohio, August 28, 1947, and the 73rd at the Wardman Park Hotel, Washington, D. C., March 4, 1948.

TENTATIVE REVISIONS OF STANDARDS

Subsequent to the 1947 Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards a proposed tentative revision of the Standard Definitions of Terms Relating to Refractories (C 71 - 47). The tentative revision was accepted by the Standards Committee on January 28, 1948, and appears in the February, 1948, Manual of A.S.T.M. Standards on Refractory Materials.

Standard Definitions of Terms Relating to Refractories (C 71 - 47).—In rounding out the list of definitions pertaining to refractories, the committee recommends the following two definitions for publication as a tentative revision:

Single-Screened Ground Refractory Material.—A refractory material that contains a natural gradation of particle sizes resulting from grinding or crushing and from which particles coarser than a specified size have been removed by screening.

Double-Screened Ground Refractory Material.—A refractory material that contains a natural gradation of particle sizes resulting from grinding or crushing and from which particles coarser and finer than two specified sizes have been removed by screening.

Standard Method of Panel Spalling Test for Fireclay Plastic Refractories (C 180 - 47).—The committee recommends the following changes for publication as a tentative revision:

Section 3(a).—Change "14" in the first line to read "15."

Section 3(c).—In the second line insert "labeled and" after "be," and add to end of the paragraph "and before the specimens have cooled to room temperature, each shall be weighed to the nearest 0.01 lb."

Section 3(d).—Add a new Paragraph (*d*) reading as follows:

(*d*) *Calculation of Fired Weight of Specimens.*—Fourteen of the 15 brick are for constructing the test panel, and the "fired" weight of each shall be calculated by use of their dried weight (Section 3 (*c*)) and per cent firing loss. The firing loss figure shall be obtained from the "extra" specimen after heating it for 5 hr. at a temperature in the range of 1900 to 3000 F., and reweighing it to the nearest 0.01 lb. The firing loss shall be expressed as percentage of the dried weight.

NOTE: Provided the combined drying and firing shrinkage of Method C 179 is to be determined simultaneously, one of the specimens for that test may be used as the "extra" brick.

Section 6 (f).—Replace with Section 5 (*f*) of Standard Method C 107, changing the last sentence to read: "The brick shall be weighed to the nearest 0.05 lb. and the loss in weight recorded as a percentage of the calculated fired weight (Section 3(*d*))."

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The modulus of rupture requirement for low duty fireclay brick, type G, in three specifications stipulates a minimum strength of 500 psi. A tentative revision increases the requirement to 800 psi. Because of not having received criticism or comments on this proposal, the committee recommends that this tentative revision be adopted as standard in the following specifications:

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

Standard Specifications for:

Refractories for Heavy Duty Boiler Service (C 64 - 47), as pertaining to Section 6(*g*).

Refractories for Incinerators (C 106 - 47) as pertaining to Section 5(*c*).

Refractories for Moderate Duty Boiler Service (C 153 - 47), as pertaining to Section 5(*b*).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (W. S. Debenham, chairman).—During the year a survey was prepared on pouring pit refractories by L. G. Ekholm of the Carnegie-Illinois Steel Corporation, and this has been incorporated in the current edition of the Manual of A.S.T.M. Standards on Refractory Materials. For the purpose of revising the present surveys and enlarging the field, the subcommittee is endeavoring to have the lead, copper, and malleable iron surveys revised by competent authors. Efforts are being made to obtain authors for surveys pertaining to the zinc and water-gas industries.

Subcommittee II on Research (R. B. Sosman, chairman).—The committee has outlined six problems relating to refractories for the purpose of encouraging further study, and these were publicized in the May, 1947, *Bulletin* of the American Ceramic Society. Written response was received on the study pertaining to the use of the spectrograph in analyzing silica brick. The speed with which certain constituents can be determined is of particular interest to both manufacturers and users of silica refractories. The method is now being used for routine determinations to learn of its reliability, particularly as compared with the usual chemical methods.

At least two universities are studying problems relating to the hot load test, as are also laboratories in the producing and consuming groups. Work is being carried out at one university in comparing P.C.E. results as obtained in gas-air *versus* acetylene-oxygen heated test furnaces.

The subcommittee recommends the investigation of the shrinkage and mechanical weakening of insulating fire brick when heated for long periods of time in hydrocarbon atmospheres and other reducing gases. Another suggestion has to do with the standardization of a testing procedure for the study of the disintegration of fireclay refractories by carbon monoxide.

Subcommittee III on Tests (S. M. Phelps, chairmen).—The committee, comprised of the chairmen of eight sections, considers any recommendation which is to be presented for action by the individual sections prior to presentation to Committee C-8. Pertinent subject matter handled by the sections was:

Section A on Load (J. D. Sullivan, chairman) sent a questionnaire containing a list of subjects for study in connection with the hot load test to all the ceramic departments of universities, which has resulted in obtaining cooperation in several instances. One laboratory is studying the effect of heating schedules and load on the flow of refractory materials. Another school is carrying out an extensive program on the possibility of developing a method for evaluating the refractories used in pouring pits. Four other universities expressed interest in cooperation, but for one reason or another they have not been able as yet to begin work.

Section B on Spalling (R. E. Birch, chairman) collected information pertaining to the suitability of an ignition loss method for obtaining the fired weight of fireclay plastic refractory brick as used in the spalling test for that material. This showed that a more convenient and accurate method could be used in place of the present procedure and, consequently, the recommended tentative revision of Standard Method C 180–47 was made.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

A problem under study relates to obtaining a more refractory mortar for the test brick than the kaolin which is now available. Mixtures of kyanite and kaolin have been tried, as well as rather pure alumina and kaolin. The subject is to be continued in an effort to obtain a material which can be removed from the test brick more readily than at present.

Another assignment pertains to a spalling procedure which might be of more specific value in testing semi-silica fireclay refractories.

Section C on Temperature (J. L. Carruthers, chairman) developed a suitable editorial footnote for Method C 24, describing compositions for cone plaque material. The principal project of the section has to do with collecting first-hand information on the variation which may result in P.C.E. determinations when made in various types of furnaces and with different methods of heating. A group of samples is to be made into pyrometric cones in a mechanical press, after which the cones will be calcined so as to reduce possible sources of error. The cones will be sent to various laboratories along with a supply of prepared cone plaque material. The results obtained should show the variation in results which may be expected from the different kinds of P.C.E. furnaces now in use.

Section E on Analysis (H. A. Heiligman, chairman) has been investigating the necessity of using absolute methyl alcohol saturated with dry hydrochloric acid gas *versus* the use of hydrochloric acid and ordinary methyl alcohol in the removal of boron when determining silica in high alumina refractories. Another subject has to do with the difficulty of slaking the sintered cake after fusion when determining alkalies of high alumina refractories. Apparently this trouble can be overcome by using a smaller amount of ammonium chloride. These features may become the subject of a revision of Standard Method C 18.

The kind of mortar used in grinding silica refractories is of importance when the material is to be analyzed for alumina. Because of the scarcity of agate mortars, those made of mullite have been used, but contamination by alumina is variable and often high, particularly since a low alumina content is the feature of some silica refractories. Efforts will be made to learn whether mortars of suitable size can be made at a reasonable cost of hard alloys or with a coating of these materials.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) has under study a panel test for evaluating shrinkage of insulating fire brick so as to make use of a method which is more in keeping with the manner in which the product is used. Plans for this investigation are under way. Another suggestion for study pertaining to the development of a shipability test for insulating brick, but the committee reported that the modulus of the product could be used as an index to their behavior during shipment.

Section G on Porosity and Permanent Volume Change (J. F. Kelly, chairman) collected information on methods for determining the permeability of refractories, which will be made available, but there is some question whether there is a sufficient need at present for a standard test to obtain this property of refractories. Consideration was given to the possibility of making use of a single heating schedule for the reheat test up to a definite temperature before branching off for the various time-temperature curves. It does not seem, however, that this can be done without affecting the results obtained.

Section H on Mortars and Plastic Refractories (R. S. Bradley, chairman).—Work carried out at the National Bureau of Standards on the study of the workability test for fireclay plastic refractories has lead to several suggestions for revision of Method C 181. These have to do with the finish of the mold, the use of a snug-fitting disk in the case of finning during the molding operation, the rate of impacts when molding the sample, and a possible basic change in the principle employed in obtaining an index to workability. The subcommittee contemplated recommended changes incorporating some of these features but believes it best to wait until a more complete study of the various features has been made so as to incorporate all changes in one tentative revision.

Section I on Disintegration by Carbon Monoxide (H. M. Kraner, chairman).—During the year a new section was added to the committee for the purpose of studying test methods pertaining to the disintegration of refractories by carbon monoxide and eventually recommending a standard procedure. The committee has made a good start in the project by delegating phases of the preliminary work to be handled by members, and these pertain to the chemistry factors as affecting the rate of action, a complete bibliography, and the preparation of a questionnaire to be sent to all laboratories carrying out the test. The latter will provide more complete information on the equipment and procedure now in use. Later, the work of the section may be directed to the effect of hydrocarbons and such features as the volatilization of silica at high temperatures in certain atmospheres.

Subcommittee IV on Heat Transfer (C. L. Norton, chairman).—Because of the involved nature of the equipment and method of testing for the thermal conductivity of refractory materials as provided for in the three Standard Methods C 182, C 201, and C 202, the subcommittee has prepared a rather extensive descriptive account to supplement the methods. This was gone over with the idea of condensing it so that it might be included in the manual as a recommended practice, but it was found that if this should be attempted, much of its practical value would be lost; consequently, the present plan is to supply a copy with each set of drawings supplied for the construction of the apparatus.

The subcommittee has been in touch with H. C. Dickinson of the Bureau of Standards, who has been developing a simplified piece of apparatus for obtaining the thermal conductivity of refractories. During a C-8 meeting Mr. Dickinson presented his equipment and described the procedure. The apparatus is small in size, and conductivity figures can be obtained at high temperatures in a relatively short time. The test sample is a 6-in. disk, $\frac{3}{8}$ to 1 in. in thickness, and is placed over the opening of a 6-in. diameter gas-fired furnace. The temperature of the heated side of the specimen is measured with an optical pyrometer and the "cold" side by the use of thermocouples cemented in a small groove. A water calorimeter of unique design is placed on top or cooler side of the specimen, and this consists of an annular guard calorimeter surrounding a circular one $2\frac{3}{4}$ in. in diameter which is used for the actual measurement. During the heating-up period, the steam generated in both calorimeters is condensed and returned to the individual units. After reaching equilibrium, the water condensed in the measuring calorimeter is by-passed into a calibrated glass container. The time required to obtain a given volume is measured, after which that water is returned to the calorimeter. Repeated measurements can be made at short intervals, and conductivity data can be obtained at high temperatures in 3 to 4 hr. after starting the test. Still more rapid testing can be carried out by keeping the furnace hot while installing other specimens. Tests show that successive determinations on the same sample have a reproducibility of about 2 per cent.

Mr. Dickinson explained that the equipment and procedure have been developed to the point where he would like to compare results with those obtained in other types of equipment. He requested cooperation by interested parties in supplying him with samples of insulating fire brick on which data from other sources have been obtained so that he could test the material and provide his results for comparative purposes. Much of the future progress to learn of the value of this simple test will depend on the response to this request.

Subcommittee V on Precision and Tolerance (W. S. Debenham, chairman) plans to suggest a proposal relating to size tolerance of brick and a method for sampling carload shipments for size measurements.

Subcommittee VI on Nomenclature (R. A. Heindl, chairman).—This subcommittee developed four definitions, two of which are submitted for approval as tentative. Definitions for chrome-magnesite brick and magnesite-chrome brick were developed during the year and were submitted to the Standards Committee in December, 1947, and were found acceptable; consequently, they were published as a tentative revision in the current edition of the Refractories Manual. Definitions for the following terms are being studied: Refractories, mullite refractories, fireclay plastic refractory, silica refractory, cast refractory (including molten cast and material formed by slip casting), castable refractory, warpage, and the simplification of items appearing under the note in the present definition for spalling.

Subcommittee VII on Specifications (G. A. Bole, chairman).—The subcommittee recommended the adoption as standard of the tentative revisions of three standard specifications. Consideration is being given to the revision of the Standard Specification for Ground Fire Clay as a Mortar for Laying Up Fireclay Brick (C 105 – 47), in which it appears that the scope could be expanded to include general-purpose clays as well as those used as a mortar.

The committee received suggestions concerning the revision of Standard Specification for Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 47 T), submitted by a manufacturer of that product. Among other things, attention will be directed to the recommended use of a flow table as specified in A.S.T.M. Method C 124 for the purpose of adjusting the amount of water to be used in preparing the castable refractories prior to molding the test specimens.

The committee received a proposed specification relating to particle size requirements for ground refractory materials, the purpose being to standardize the method of specifying particle size requirements and tolerances for single- and double-screened ground materials. Because the proposal does not involve screen sizes to be used, a feature which is to be specified by the purchaser, the subject matter will be turned over to the Subcommittee on Classifications for consideration as a classification.

Subcommittee VIII, Editorial (L. J. Trostel, chairman).—This subcommittee has been active during the year in preparing subject matter for the current edition of the C-8 Manual on Refractories. This has become the most important publication on refractories in the field of specifications, classifications, definitions, methods of testing, and general literature pertaining to the properties and use of refractory materials in a number of industries. The extent of its distribution is indicated by the fact that the number of copies printed is the second highest of the manuals printed by the Society.

Because of the expanding use of the A.S.T.M. terminology for classes of refractories, and as an aid in promoting this, action was taken to shorten the titles of the high, intermediate, and low heat duty classes. This resulted in deleting the word "heat" in the respective titles. Unfortunately, this editorial change was made after printing the manual, but efforts are to be made to have the shorter name introduced for current use.

Subcommittee IX on Classifications (S. M. Swain, chairman).—Subcommittee IX has given considerable thought to recommendations received in connection with the revision of Standard Classification C 27, but after deliberation it was of the opinion that no change should be made. The subcommittee has under consideration at present a classification for high-fired super duty fireclay refractories, for semisilica fireclay material, and the classification pertaining to ground refractory materials mentioned under the Subcommittee on Specifications.

Subcommittee X on Petrography (H. M. Kraner, chairman).—This subcommittee's work to date has been published in the new Manual as a Suggested Practice for Petrographic Techniques for the Study of Refractories.

Subcommittee XI on Special Refractories (M. C. Booze, chairman).—The subcommittee reports that its work is handicapped because of the need for additional methods of testing, which are needed for the evaluation of special refractories. Because the field of refractories and the scope of work of the committee are becoming larger, the suggestion has been made that perhaps the membership of Committee C-8 should be made larger so as to cover more ground without the overlapping of members on subcommittees and sections.

Subcommittee XII on Carbon Refractories (L. A. Smith, chairman).—This new subcommittee has been formed to cover work in the field of carbon refractories as such and products which contain carbon, including graphite. One of the principal problems will relate to the use of carbon refractories in blast furnaces. At present the personnel for the subcommittee is being completed.

Subcommittee XIII on Foreign Tests and Specifications (J. F. Kelly, chairman).—This new subcommittee was formed for the purpose of keeping in touch with developments in foreign countries which would be of interest to Committee C-8. For example, literature has been received from the Mexican Bureau of Standards which shows that its specifications and methods of test are patterned after C-8 literature. Similar subject matter has been received from the South African Government and this is being reviewed. Furthermore, recently the British have organized a committee for the purpose of investigating standards relating to refractories, and the minutes of the first meeting will be turned over to the new subcommittee.

The Nominating Committee's slate (L. C. Hewitt, chairman) was presented, and the following candidates were elected unanimously for the ensuing term of two years:

Chairman, R. B. Sosman
Vice-Chairman, R. S. Bradley
Secretary, W. R. Kerr

This report has been submitted to letter ballot of the committee, which consists of 37 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.

L. J. TROSTEL,
Vice-Chairman.

S. M. PHELPS,
Secretary.

REPORT OF COMMITTEE C-9*

ON

CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates has held two meetings during the year: on June 16, 1947, during the Annual Meeting of the Society in Atlantic City, N. J., and on November 5, 1947, in Chicago, Ill.

During 1948 the committee suffered the loss through death of J. E. Boyd and Joseph C. Pearson. Mr. Boyd, a member of Committee C-9 since 1938 and of the Society since 1937, was sales engineer with Weston & Brooker Co. He had formerly been Materials Engineer of the Georgia State Highway Department. At the time of his death Mr. Boyd was a member of Subcommittee III-e on Methods of Testing Concrete Aggregates for Physical Characteristics.

With the death of Joseph C. Pearson on March 16, 1948, Committee C-9 lost one of its oldest members in point of service. He had been a member of the committee since 1919 and served it as secretary from 1922 to 1926. At the time of his death Mr. Pearson was a member of the Executive Committee and of Subcommittee II-a on Evaluation of Data. On December 31, 1947, Mr. Pearson retired as Director of Research for the Lehigh Portland Cement Co. after 24 years service. He had previously been with the National Bureau of Standards for 14 years.

Concurrently with Mr. Pearson's retirement a petition was circulated among members of Committee C-9 nominating him for honorary membership in accordance with procedures outlined in the committee's Regulations. Death overtook Mr. Pearson before the Executive Committee had an opportunity to act on the petition.

Sanford E. Thompson Award.—This year's Sanford E. Thompson Award for a paper of outstanding merit on concrete and concrete aggregates presented before the Society will be made to Mr. W. C. Hanna, Chief Chemist and Chemical Engineer, California Portand Cement Co. for his paper on "Unfavorable Chemical Reactions of Aggregates in Concrete and a Suggested Corrective," published in the 1947 *Proceedings*.[1] This is the seventh Sanford E. Thompson Award, previous ones having been made in 1947, 1946, 1944, 1941, 1940 and 1939.

Honorary Membership.—In accordance with provisions in the committee's Regulations, Sanford E. Thompson was elected an Honorary Member of Committee C-9. Mr. Thompson has contributed to the progress of the committee since its inception. He served as its first chairman and held that position from 1914 to 1922. The election to honorary membership constitutes grateful recognition to Mr. Thompson for his long and valuable services.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, K. B. Woods.
Vice-Chairman, Fred Hubbard.
Secretary, Stanton Walker.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard:

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 986 (1947).

Tentative Specifications for Ready-Mixed Concrete (**C 94 – 47 T**)[2] without revision.

Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (**C 125 – 46 T**)[3] with the following revision of the definition of the term "admixture:"

> *Admixture.*—A material other than water, aggregates and portland cement (including air-entraining portland cement and portland blast-furnace slag cement) that is used as an ingredient of concrete and is added to the batch immediately before or during its mixing.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in two of its standards, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Method of Test for Organic Impurities in Sands for Concrete (**C 40 – 33**):[3]

Section 5.—Delete Paragraph (*b*) and the accompanying Color Plate I for Colors of Treated Sands with Suggested Ranges of Application. Renumber Paragraph (*a*) as Section 5.

Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (**C 117 – 37**).[3]—This method is under the joint jurisdiction of Committee D-4 on Road and Paving Materials and Committee C-9. The following revision, substantially editorial in nature, is recommended, subject to concurrence of Committee D-4:

Section 2(a).—Change to read as follows:

> (*a*) *Sieves.*—A nest of two sieves, the lower being a No. 200 (74-micron) sieve and the upper approximately a No. 16 (1190-micron) sieve, both conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives which have stood without revision for two or more years be retained as tentative:

Tentative Methods of Tests for:[3]

Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 46 T),
Water Retention Efficiency of Methods for Curing Concrete (C 156 – 44 T),
Air Content (Volumetric) of Freshly Mixed Concrete (C 173 – 42 T), and

Tentative Specifications for:[3]

Waterproof Paper for Curing Concrete (C 171 – 42 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Committee C-9 completed a year of operation under the reorganized subcommittee structure referred to in its 1947 Report.[5] A large portion of the effort expended by the new subcommittees to date has, of necessity, been of an organizational nature. The activities of the committee are divided into three groups: Administration, Research, and Specifications and Test Methods.

Administration, under the guidance of Vice-Chairman Fred Hubbard, includes subcommittees on finance, papers and symposia, Sanford E. Thompson Award, and editorial and definitions. The Subcommittee on Papers and Symposia has arranged for a Symposium on

---

[2] 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[3] 1946 Book of A.S.T.M. Standards, Part II.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 246 (1947).

Procedures for Identifying Reactive Materials, for the Fifty-First Annual Meeting of the Society. The Editorial and Definitions Subcommittee has recommended the adoption as standard of the Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 – 46 T) with a revision in one definition as referred to earlier in this report. It also has under consideration a revision of the Tentative Definition of the Term Aggregate (C 58 – 28 T).

Group II, Research, is under the chairmanship of A. T. Goldbeck. It includes subcommittees on evaluation of data, chemical reactions of aggregates, elasticity and volume changes, durability, dynamic testing, and mineralogical characteristics of aggregates.

Subcommittee II-a plans to supplement the A.S.T.M. Manual on Presentation of Data by simple explanations and examples using data of tests of concrete. Furthermore, an attempt will be made to develop information on methods for calculating probability, estimating 28-day strengths of concrete from early tests, determining probable error of estimate, identifying and discarding defective results, determining number of tests necessary to represent a "population," and obtaining curve of "best fit" for plotted data.

Subcommittee II-b on Chemical Reactions of Aggregate in Concrete is among the more active subcommittees. Three meetings have been held with a high percentage of the membership and large numbers of visitors in attendance. The meetings have generally consisted of formal papers on test methods for identifying reactive materials followed by lively discussions on the problem.

The broad field of durability of concrete has been assigned to Subcommittee II-d. A study of the literature on test methods used to date and variables involved is being made prior to undertaking future fundamental studies. Subcommittee II-f is preparing a list of rock types likely to be of most interest to the engineer together with descriptions and methods for identification. It is hoped to supplement the list at some time in the future with a discussion of the relative quality of each rock type as an aggregate for concrete.

Group III, Specifications and Test Methods, under the chairmanship of R. R. Litehiser, includes eleven subcommittees. Of these, six are devoted to test methods and five to specifications as well as test methods.

The subcommittees dealing with methods of testing properties of concrete have under consideration such procedures as measurement of entrained air by pressure methods, measurement of bleeding, and evaluation of bond particularly as affected by admixtures. In addition, they are reviewing existing routine methods.

Subcommittee III-d on Specifications for Aggregate has as a principal activity the coordination of recommendations of the A.S.T.M. and the American Association of State Highway Officials. It has also suggested for the consideration of other subcommittees a number of methods of test and the need for revision in certain existing test methods.

Other matters receiving consideration which may be mentioned briefly are: a major revision in the Standard Specifications for Lightweight Aggregates for Concrete (C 130 – 42); a survey of test methods and specifications for curing compounds with the view of preparing new standards; and studies of admixtures. In connection with the subject of admixtures attention is directed to the Proposed Method for Evaluation of Air-Entraining Admixtures for Concrete published as information in the ASTM Bulletin.[6]

---

[6] ASTM Bulletin, No. 148, October, 1947, p. 29.

This report has been submitted to letter ballot of the committee, which consists of 74 voting members including 4 honorary members having voting privileges; 70 members returned their ballots, of whom 67 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
Chairman.

STANTON WALKER,
Secretary.

T

REPORT OF COMMITTEE C-11*
ON
GYPSUM

The activities of Committee C-11 on Gypsum were practically discontinued during the war. A meeting held in conjunction with the Annual Meeting of the Society in June, 1946, was not particularly well attended nor was a special meeting of the committee held on May 9, 1947, in Washington, D. C. Through correspondence, efforts were made during the past year to develop renewed activity. As a result, the committee met on March 2, 1948, in Washington with an attendance of twelve members and two alternates out of a membership of nineteen, with five guests present. No new members have been added, but the representation of many of the organizations on the committee has been changed. In addition, four members have resigned. The committee now consists of nineteen members, of whom four are classified as consumer, eight as producer and seven as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. S. Wells.
Vice-Chairman, H. F. Gardner.
Secretary, C. E. Abbey

The entire subcommittee structure of the committee is to be revamped in the ensuing months.

ADOPTION OF TENTATIVE AS STANDARD

Tentative Definitions of Terms Relating to Gypsum (**C 11 - 41 T**).—Committee C-11 recommends that these tentative definitions be approved for reference to letter ballot of the Society for adoption as standard without revision. Upon adoption, these definitions are to be incorporated in the Standard Definitions of Terms Relating to Gypsum (C 11 - 41).

WITHDRAWAL OF STANDARDS

Standard Specifications for Calcined Gypsum for Dental Plasters (**C 72 - 40**) and *Standard Specifications for Gypsum Pottery Plaster* (**C 60 - 40**).—The committee recommends these specifications be withdrawn as these standards cover material for which users generally write their own specifications without regard to a common standard, making it difficult for Committee C-11 to write an acceptable revision of the present standards which are definitely not correct.

WITHDRAWAL OF TENTATIVE REVISIONS

The committee also recommends that the tentative revision of the Standard Methods of Testing Gypsum and Gypsum Products (C 26 - 42) which was submitted in June, 1942, be withdrawn, as Section 19, to which this revision referred, needs further revision.

The committee also recommends that the tentative revision of Standard Specifications C 72 be withdrawn since the standard itself is being discontinued.

TENTATIVE REVISION OF STANDARD

Standard Specifications for Gypsum

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

Plasters (**C 28 – 40**).—Committee C-11 recommends the following tentative revision of these specifications in order to change the strength requirements for gypsum plasters to dry compressive strength rather than the present tensile strength:

Section 4.—Change this section to read as follows:

> 4. Gypsum ready-sanded plaster shall contain not more than 3 parts of sand to 1 part of gypsum by weight. The gypsum shall contain not less than 60.5 per cent by weight of calcined gypsum. The remainder may consist of fiber, materials to control setting time and working qualities, and inert fines.

Section 6.—Revise to read as follows:

> 6. *Compressive Strength.*—Gypsum ready-sanded plaster shall have a compressive strength of not less than 500 psi.

Section 10.—Revise to read as follows:

> 10. *Compressive Strength.*—Gypsum neat plaster as tested with two parts of standard sand shall have a compressive strength of not less than 750 psi.

Section 14.—Revise to read as follows:

> 14. *Compressive Strength.*—Gypsum wood fibered plaster shall have a compressive strength of not less than 1200 psi.

Section 18.—Revise to read as follows:

> 18. *Compressive Strength.*—Calcined gypsum for finishing coats shall have a compressive strength of not less than 1200 psi.

These tentative revisions are made in the interest of ease of testing and also due to the lack of acceptance of the present method as giving a true tensile strength. The new compressive strength requirements raise the minimum strength substantially over the previous limits either tensile or compressive. This will involve further detailed changes which will be included in an over-all study of the Standard Methods of Testing Gypsum and Gypsum Products (C 26). There is additional study under way by the committee of changes of a minor nature in other A.S.T.M. standards for gypsum and the committee is also making recommendations to the Federal Specification Board on Federal specifications covering gypsum products.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

This report has been submitted to letter ballot of the committee, which consists of 19 members; 19 members have returned their ballots, of whom 15 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

L. H. YEAGER,
Secretary.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE C-12*
ON
MORTARS FOR UNIT MASONRY

Committee C-12 on Mortar for Unit Masonry held two meetings during the past year: on June 17, 1947, in Atlantic City, N. J., and on March 3, 1948, in Washington, D. C. Progress was reported in the activities of all subcommittees.

At the present time, the committee consists of 35 members, of whom 14 are classified as producers, 8 as consumers, and 13 as general interest members. There are 5 consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Theodore I. Coe
First Vice-Chairman, J. W. Whittemore
Second Vice-Chairman, H. D. Baylor
Secretary, J. A. Lee.

CONTINUATION OF TENTATIVE WITHOUT REVISION

The committee recommends that the Tentative Specifications for Mortar for Reinforced Brick Masonry (C 161 – 44 T) be retained as tentative without revision. The reason for this recommendation is that, after the proposed Tentative Specifications for Mortar for Unit Masonry has stood for one year, it will be desirable either to withdraw the present Specifications C 161 or to amend it to conform to the requirements of the general mortar specifications.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (F. O. Anderegg, chairman) has developed a proposed Method of Analyzing Fresh Mortar, that is, a mortar as used by the mason taken from the construction site. This method has been submitted to the Society for publication as information only.

This subcommittee has also reported the results of an investigation of various waterproofing treatments for masonry walls which Mr. Anderegg will present in a paper entitled "Testing Surface Waterproofers,"[1] at the 1948 Annual Meeting of the Society.

Subcommittee III on Specifications and Methods of Test for Mortar (P. M. Woodworth, chairman) has prepared proposed Tentative Specifications for Mortar for Unit Masonry. These specifications are the result of research and discussion by members of the committee extending over a period of about ten years. This proposed tentative after approval of further revisions by the committee will be submitted to the Administrative Committee on Standards during the coming year.

Working Subcommittee on Efflorescence (P. L. Rogers, chairman).—Members of this subcommittee are investigating methods of determining efflorescence tendencies of mortars. Progress reports of these studies have been made to the committee.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 20 members returned their ballots, of whom 19 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

THEODORE I. COE,
Chairman.

HARRY C. PLUMMER,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] ASTM BULLETIN, No. 156, January, 1949, p. 71 (TP33).

REPORT OF COMMITTEE C-14*
ON
GLASS AND GLASS PRODUCTS

Committee C-14 on Glass and Glass Products held one meeting during the year in Chicago, Ill., on April 28, 1948, at the time of the annual meeting of the American Ceramic Society.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. A. Wadman.
Vice-Chairman, E. W. Tillotson.
Secretary, F. V. Tooley.

A new subcommittee on illuminating glassware has been proposed but no action will be taken pending further information as to the extent of coverage in this field by the Sectional Committee on Optics functioning under the procedure of the American Standards Association.

Activities of Subcommittees

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman). —A glossary of glass factory terms is essentially complete. Definitions of some fifteen technical terms approved by the Glass Division of the American Ceramic Society will be submitted to letter ballot of the committee for publication as tentative.

Subcommittee III on Chemical Properties (J. F. Greene, chairman).—Durability tests are in progress intended to cover circumstances of service.

Subcommittee VI on Structural Glass (A. H. Baker, chairman).—Methods for testing cellular glass blocks have been formulated and submitted to letter ballot of the committee for publication as tentative.

Subcommittee VII on Glass Containers (K. C. Lyon, chairman).—Revisions of three standards, namely, Method of Hydrostatic Pressure Test on Glass Containers (C 147 – 43), Method of Polariscopic Examination of Glass Containers (C 148 – 43), and Method of Thermal Shock Test on Glass Containers (C 149 - 43), have been prepared and will be submitted to letter ballot of the committee. Studies are being made on impact tests.

Subcommittee VII-A on Pharmaceutical Containers (F. C. Flint, chairman).—After extensive study, three test methods for durability of glass containers have been prepared for submission to letter ballot of the committee. These three methods involve autoclave tests at 121 C. on 2-oz. and 16-oz. bottles attacked by water and acid, respectively, and for powdered glass.

This report has been submitted to letter ballot of the committee, which consists of 58 members; 25 members returned their ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

Louis Navias,
Chairman.

S. R. Scholes,
Secretary

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

REPORT OF COMMITTEE C-16*
ON
THERMAL INSULATING MATERIALS

During the past year, Committee C-16 on Thermal Insulating Materials held two meetings: at State College, Pa., on November 3 to 5, 1947, and at Washington, D. C. on March 1 to 3, 1948. At the business meeting of the committee on March 3, 1948, election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Ray Thomas.
Vice-Chairman, B. A. Hollenbeck.
Secretary, W. L. Scott.
Advisory Committee, C. B. Bradley, E. R. Queer, and K. M. Ritchie.

The committee was saddened by the death of J. H. Walker, its first chairman, on December 1, 1947. A memorial resolution expressing the committee's sorrow was prepared and inscribed in the permanent records of the committee. A copy of the resolution was also sent to Mr. Walker's family.

In recognition of its first ten years of existence, a history of the organization and activities of the committee from 1938 to 1948, inclusive, was presented at the Washington meeting by C. B. Bradley, appointed historian for the purpose.

In the interest of more effective operation, a plan for reorganization and realignment of the executive direction and supervision of the subcommittee activities was presented at the Washington meeting. Amendments to the by-laws necessary to accomplish this were given a first reading for consideration by the committee.

The committee now consists of 76 members, of whom 56 are voting members; 25 are classified as producers, 18 as consumers, and 13 as general interest members.

REVISION OF TENTATIVE

Tentative Method of Test for Water Vapor Permeability of Sheet Materials Used in Connection with Thermal Insulation (**C 214 - 47 T**).—The committee recommends that this method be revised as appended hereto.[1] The revision consists principally of the addition of an alternate water method (Method B) and the inclusion of requirements covering certain details not previously specified.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends revisions of the following three standards for publication as tentative revisions:

Standard Method of Test for Bulk Density of Thermal Insulating Cement (**C 164 - 44**):

Title and Section 1.—Change "bulk density" to read "storage density."

Section 2.—Change to read as follows: "2. The storage density of an insulating cement is the weight per unit volume of the packaged cement in packages as received, expressed as pounds per cubic foot of storage space required."

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] This revised method was accepted by the Society and appears in 1948 Supplement to Book of A.S.T.M. Standards, Part II.

Section 5.—Change "bulk density" to read "storage density" and revise the formula as follows:

$$\text{Storage density} = \frac{M}{w \times h \times l}$$

Standard Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (**C 166 - 45**):

Section 2.—Change to read as follows:

2. (*a*) *Wet Covering Capacity.*—The wet covering capacity of an insulating cement is the volume occupied in "square feet 1 in. in thickness per 100 lb. of dry cement" when the cement is mixed with the recommended amount of water and molded in accordance with Section 5.

(*b*) *Dry Covering Capacity.*—The dry covering capacity of an insulating cement is the volume occupied in "square feet 1 in. in thickness per 100 lb. of dry cement" after the wet cement has been molded and dried to constant weight in accordance with Section 5.

(*c*) *Volume Change upon Drying.*—The volume change upon drying is the percentage change in volume of the wet molded cement that occurs when the dry cement is mixed with the recommended amount of water, molded and dried to constant weight in accordance with Section 5.

Section 6.—Change the units of expression for "C_w" and "C_d" to read as follows:

C_w = wet covering capacity in square feet 1 in. in thickness per 100 lb. of dry cement,

C_d = dry covering capacity in square feet 1 in. in thickness per 100 lb. of dry cement.

Standard Definitions of Terms Relating to Thermal Insulating Materials (**C 168 - 47**):

Make the following revisions in five definitions:

Storage Density of Thermal Insulating Cement.—The weight per unit volume of the packaged cement in packages as received, expressed as pounds per cubic foot of storage space required.

Dry Covering Capacity of Thermal Insulating Cement.—The volume occupied in "square feet 1 in. in thickness per 100 lb. of dry cement" after the wet cement has been molded and dried to constant weight.

Thermal Insulating Cement.—A prepared composition, in dry form, comprising granular, flaky, fibrous, or powdery materials, which when mixed with a suitable proportion of water to develop a plastic consistency, and applied and dried in place, forms a coherent covering that affords substantial resistance to heat transmission.

Volume Change upon Drying of Thermal Insulating Cement.—The percentage change in volume of the wet molded cement that occurs when the dry cement is mixed with the recommended amount of water, molded and dried to constant weight.

Wet Covering Capacity of Thermal Insulating Cement.—The volume occupied in "square feet 1 in. in thickness per 100 lb. of dry cement" when the cement is mixed with the recommended amount of water and molded.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the new definition for molded-type pipe insulation which was submitted in June, 1947, as a tentative revision be adopted as standard and incorporated in the Standard Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 47).

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following six tentative specifications and one method be approved for reference to letter ballot of the Society for adoption as standard, with revisions in six as indicated:

Tentative Specifications for Structural Insulating Board Made from Vegetable Fibers (**C 208 - 46 T**), without revision,

Tentative Specifications for 85 per cent Magnesia Thermal Insulating Cement (**C 193 - 44 T**), revised as follows:

Section 1.—Change the temperature in the last line from "200" to read "100" F.

Section 3.—Under the requirements for

physical properties change "Bulk density, cu. ft. per 100 lb." to read "Storage density, lb. per cu. ft." and change "Dry covering capacity, min., bd. ft. per 100 lb. of dry cement" to read "Dry covering capacity, min., sq. ft. 1 in. in thickness per 100 lb. of dry cement."

Section 5 (b).—Change "bulk density" to read "storage density" in two places.

Tentative Specifications for Asbestos Thermal Insulating Cement (**C 194 – 44 T**), revised as follows:

Section 1.—Change the temperature in the last line from "140" to read "100" F.

Section 3.—Make the same change as recommended above for Tentative Specifications C 193.

Section 5 (a).—Change "Bulk density" to read "Storage density" in two places.

Tentative Specifications for Mineral Wool Thermal Insulating Cement (**C 195 – 47 T**) revised as follows:

Section 1.—Change the temperature in the last line from "140" to read "100" F.

Section 3.—Make the same change as recommended above for Tentative Specifications C 193. In addition, change the thermal conductivity maximum limit at mean temperature of 500 F. from "0.90" to read "0.85" Btu. This decrease in the specification maximum limit for thermal conductivity was recommended as a better representation of commercial practice.

Section 5 (a).—Change "bulk density" to read "storage density" in two places.

Tentative Specifications for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (**C 196 – 47 T**), revised as follows:

Section 1.—Change the temperature in the last line from "140" to read "100" F.

Section 3.—Make the same change as recommended above for Tentative Specifications C 193.

Section 5 (a).—Change "bulk density" to read "storage density" in two places.

Tentative Specifications for Diatomaceous Silica Thermal Insulating Cement (**C 197 – 47 T**), revised as follows:

Section 1.—Change the temperature in the last line from "600" to read "100" F.

Section 3.—Make the same change as recommended above for Tentative Specifications C 193.

Section 5 (a).—Change "bulk density" to read "storage density" in two places.

Tentative Methods of Testing Structural Insulating Board Made from Vegetable Fibers (**C 209 – 46 T**), revised as follows to improve accuracy and precision:

Section 8.—Change the speed of testing from "12 ± 2" to "6 ± 2" in. per min.

Section 13.—Change the rate of separation of the jaws of the testing machine from "2" to read "2 ± $\frac{1}{4}$" in. per min.

Section 17.—Delete the second sentence and substitute the following: "After the adhesive has set a sufficient length of time, the specimen shall be attached by means of the hooks to the jaws of the testing machine. The rate of separation of the jaws shall be set at 2 ± $\frac{1}{4}$ in. per min. and a load shall then be applied at the hooks until separation occurs."

Section 20.—Delete the second sentence because a specimen smaller than 12 by 12 in. under certain circumstances is no longer permitted.

Section 21.—In the second sentence, insert the phrase "with reasonable accuracy" after the word "measured."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Flexural Strength of Preformed Block Type Thermal Insulation (C 203 – 45 T) be continued as tentative without revision as the Subsection on Handleability has not completed its consideration of this and other methods of test.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

One phase of the committee's work that should be mentioned is that covered by Subcommittee IX on Vapor Barriers for Insulation. The specifications adopted and proposed by this subcommittee have been questioned by other committees and organizations who are also interested in the moisture-resistant characteristics of materials with which they are dealing. In some cases there is some conflict of thought as to procedure and result, and a feeling that a common test should satisfy all concerned. This is desirable, but other available methods do not accommodate the demands of the services covered by the committee and much work remains to be done before a common test is possible. In the meantime Committee C-16 needs something to guide its members and others similarly interested. Although, some existing methods do not satisfy the requirements of Committee C-16, an effort will be made, assisted by the newly-formed Planning Subcommittee, to work out a general test procedure, with appropriate modifications where necessary, that will eventually be generally adaptable.

This report has been submitted to letter ballot of the committee, which consists of 56 voting members; 53 members returned their ballots, 52 of whom have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

RAY THOMAS,
Chairman.

K. M. RITCHIE,
Secretary.

The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE C-18[*]

ON

NATURAL BUILDING STONES

Committee C-18 on Natural Building Stones held a meeting in Washington, D. C., on March 2, 1948, which was preceded by meetings of the subcommittees.

The committee records with sorrow the death of C. L. Van Fleet, consulting member for the past two years. Mr. Van Fleet has long been editor of the magazine "Stone" and his work on advancing the quality of architectural materials will be long remembered.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. W. Currier.
Vice-Chairman, T. I. Coe.
Secretary, F. S. Eaton.

D. W. Kessler was appointed as the representative of the Society on the American Standards Association project for formulating a specification on granite.

NEW TENTATIVES

The committee recommends the following two methods and the definitions of several types of building stones, as appended to this report,[1] for publication as tentative:

Methods of Test for:[1]
Durability of Slate for Roofing,
Combined Effect of Temperature Cycles and Weak Salt Solutions, and
Definitions of:[1]
Terms Relating to Natural Building Stones.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends revisions of the following standard definition and two methods of test, as indicated, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Definition of the Term Slate (**C 119 – 27**):

Change the definition and Note 1 to read as follows by the addition of the italicized words and the omission of those in brackets:

Slate.—A microgranular *metamorphic rock* [crystalline stone] derived from argillaceous sediments [by regional metamorphism], and characterized by *an excellent parallel* [a perfect] cleavage entirely independent of original bedding, *by which cleavage the rock may be split easily into relatively thin slabs* [which cleavage has been induced by pressure within the earth.]

NOTE 1.—The essential mineral constituents are white mica (chiefly sericite) and quartz. [Prominent] Accessory constituents *may be* [are] black mica (biotite), chlorite, [and] hematite, [Minor accessory constituents are] carbonates, magnetite, apatite, clay, andalusite, barite, rutile, pyrite, [graphite], feldspar, zircon, tourmaline, and *graphite or other* carbonaceous matter.

NOTE 2.—Igneous slates because of their rare occurrence and insignificant commercial importance are not covered in this definition.

Standard Method of Test for Water Absorption of Slate (**C 121 – 31**):

Section 4 (*a*).—Change the present requirement for temperature of the drying oven from "110 ± 3 C." to "105 ± 2 C."

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] These methods and definitions were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

Section 4 (c).—Change to read as follows: "(*c*) The specimens shall be entirely immersed in filtered or distilled water at approximately 20 ± 5 C. for 48 hr., then removed one at a time, the surface wiped dry with a slightly damp, absorbent towel and each specimen immediately weighed to the nearest 0.01 g."

In the second line of the note following this section change the word "cloth" to "towel."

Section 6 (a).—Change the symbols in the formula for calculating absorption to read as follows:

$$\text{Absorption, per cent} = \frac{W_2 - W_1}{W_1} \times 100$$

This change is recommended to avoid conflicting symbols in this test and in the test for durability which is being offered as a new tentative.

Standard Methods of Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (**C 120 - 31**):

Section 4.—Change the drying temperature from "110 ± 3 C." to "105 ± 2 C."

Section 6.—Change the third sentence to read as follows: "The load shall be applied perpendicular to the planes of cleavage through a third knife-edge at the middle of the span at a rate of approximately 100 lb. per min."

Section 7.—Change to read as follows:

7. The modulus of rupture shall be calculated as follows:

$$R = \frac{3Wl}{2bd^2}$$

where:

R = modulus of rupture in pounds per square inch,

W = breaking load in pounds,

l = span length between supporting knife-edges in inches,

b = width of specimen at the center in inches, and

d = thickness of the specimen at the center in inches.

Section 8.—Change the first two sentences to read as follows: "The average of all modulus of rupture values for specimens cut parallel to the grain from structural or electrical slate or parallel to the long dimension of the shingle in the case of roofing slate, shall be reported as the modulus of rupture 'across the grain.' The average for specimens cut perpendicular to the grain shall be reported as the modulus of rupture 'with the grain'."

Section 11.—Change the indentification of the symbols in the formula from "W'" and "Δ" to read as follows:

W' = load coordinate of the point in pounds,

Δ = deformation coordinate of the point in inches.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman) has made a study, in connection with the formulation of definitions, of 25 varieties of stone which are extensively used in masonry of various kinds. It was found to be impossible to formulate adequate definitions for granite and marble that would include all of the products which have, by long-established practice, been classed under these headings. For this reason the subcommittee has decided to offer scientific definitions and commercial definitions for both types. The latter will be less restrictive and permit serpentines and most of the travertines to be classed as marbles, while the term granite will include, in addition to the true granites, such related materials as gneiss, granite gneiss, syenite, monzonite, granodiorite, and intermediate varieties.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Because the limestones and marbles merge into each other, it was hoped to find some satisfactory basis for distinguishing between the borderline individual materials. The commonly used criterion has been the degree of polish that can be obtained on the stone. This basis is not very definite because some of the materials commonly classed as limestones will take nearly as good a polish as other materials that are, by established practice, rated as marbles. It seems, therefore, that a thoroughly satisfactory delineation between the two types will have to be deferred until a more restrictive definition for polish and a test for degree of polish can be found.

A definition for texture is offered in the new Tentative Definitions of Terms Relating to Natural Building Stones.[1] Considerable time has been spent in an attempt to formulate definitions for various surface finishes. This subject is quite involved because the finishes differ considerably for different types of stone. It seems desirable to defer the definitions on surface finishes until the work can be correlated with that of a Subcommittee of a Committee on Specifications for Granite functioning under the American Standards Association.

Subcommittee III on Test Procedures (D. W. Kessler, chairman) has conducted considerable research for the purpose of finding a suitable durability test for the very dense, crystalline stones. Granites and marbles have very low porosity, and past studies have indicated that frost action plays no appreciable part in the weathering of such materials. Due to the fact that some individual materials in each type have shown unsatisfactory durability under service conditions, it was decided to investigate thermal effects as a weathering agent. The work was based on the assumption that thermal expansion of the individual crystals making up the stone, may cause deterioration, for example, in marble because calcite has both positive and negative coefficients, depending on the axial direction.

In granite there are two or more prominent minerals which have positive coefficients in all axial directions, but the coefficients differ considerably. The studies have been made by subjecting specimens to temperature cycles (25 to 105 C.) and measuring the permanent increases in volume and absorption as indicators of the deterioration. The values for different marbles show appreciable differences in deterioration rates which appear to correspond to their observed weathering rates in structures. Since there are probably always appreciable amounts of water-soluble salts in masonry that can migrate under moist conditions and form crystals in the pores, it was decided to determine the effect of weak salt solutions and temperature cycles together. Specimens were placed in shallow dishes with small amounts of gypsum and water. These were heated to 105 C. until the water evaporated, then cooled, after which more water was added and the cycle repeated. As the gypsum has a low solubility and there was always an excess of this, the strength of solution was reasonably well controlled at about 1 part of salt to 450 parts of water. This procedure causes marked increases in rates of volume and absorption changes, and the results on various marbles are being studied in relation to their rates of weathering in structures. The correlations have been sufficiently encouraging to justify offering the procedure as a tentative method of test for durability.

Studies were made to determine if the absorption test could be used in lieu of the "depth of softening" as a measure of the deterioration of slate in the sulfuric acid test. The determination of the "depth of softening" as used in the Federal Specification for Roofing Slate has been found difficult to standardize. Both tests were applied to a representative group of samples, and the agreement of results was satisfactory. A new Method of Test for Durability of Slate for Roofing[1] is proposed for publication, as tentative.

Subcommittee IV on Specifications (W. C. Clark, chairman) has under consideration two specifications, one for roofing slate and one for marble. The one on roofing slate is similar to the Federal Specification on this product except for the means of determining durability.

Subcommittee V on Research (J. R. Shank, chairman), which was delegated to study stone-setting mortars, has looked into the question as to whether this is a proper subject to be undertaken by the committee. It will be discussed with Committee C-12 on Mortar for Unit Masonry. Although it is admitted to be a problem of greatest interest in line with stone construction, the consensus was that it does not fall within the scope of Committee C-18. Two problems in connection with roofing slate, namely the development of a test that will classify slates with respect to their susceptibility to breakage on the roof and a test to determine the permanence of color in green slates are under consideration, but both of these will require considerable research work and to date the necessary time and funds have not been available.

This report has been submitted to letter ballot of the committee, which consists of 19 voting members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

OLIVER BOWLES,
Chairman.

D. W. KESSLER,
Secretary.

REPORT OF COMMITTEE D-1*

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: in Atlantic City, N. J., June 18 to 20, 1947, in connection with the Annual Meeting of the Society, and in Washington, D. C., March 1 to 3, 1948, in connection with the Spring Group Committee Meetings.

At the June, 1947, meeting the following two papers were presented: "The Gloss Measurement of Paint Finishes," by Richard S. Hunter, of the Henry A. Gardner Laboratory, and a "Report on the Evaluation of Surface Treatment of Steel Prior to Painting,"[1] by Arnold J. Eickhoff, of the National Lead Co., Research Laboratories.

At the March, 1948, meeting the following two papers were presented: "The Measurement of Transparency of Surface Coatings,"[2] by Roy Kienle, J. Gross, L. A. Melsheimer and E. I. Stearns, Calco Chemical Division, American Cyanamid Co., and "A Method of Testing for Hiding Power,"[2] by Fred Schmutz and J. Gallagher, New Jersey Zinc Co.

During the past year a new Subcommittee XII on Exterior Exposure Testing of Paints on Wood has been organized under the chairmanship of Wayne Fuller.

The D-1 Membership Committee, which was organized in 1946 to obtain new members for Committee D-1, particularly members engaged directly in the protective coating industry, has been very active. The Membership Committee has succeeded in adding 22 new members to Committee D-1.

The D-1 Inter-committee Relations Committee which is composed of the D-1 representatives on other technical committees is continuing to follow closely the activities of other technical committees which may be of interest to Committee D-1.

Cooperative work with the Federation of Paint and Varnish Production Clubs has continued active during the year under the Joint Committee (Milton Glaser, chairman). The Federation has now approved the following eight A.S.T.M. standards and assigned the Federation numbers indicated:

Standard Methods of:

Test for Tinting Strength of White Pigments (A.S.T.M. D 332 - 36; FPVPC No. 1-32),

Test for Mass Color and Tinting Strength of Color Pigments (A.S.T.M. D 387 - 36; FPVPC No. 2-32),

Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (A.S.T.M. D 610 - 43; FPVPC No. 3-39),

Evaluating Degree of Resistance to Chalking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. D 659 - 44; FPVPC No. 4-39),

Evaluating Degree of Resistance to Checking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. D 660 - 44; FPVPC No. 5-39),

Evaluating Degree of Resistance to Cracking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. D 661 - 44; FPVPC No. 6-39),

Evaluating Degree of Resistance to Erosion of Exterior Paints of the Linseed-Oil Type (A.S.T.M. D 662 - 44; FPVPC No. 7-39),

Evaluating Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed-Oil Type (A.S.T.M. D 772 - 47; FPVPC No. 8-39).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] ASTM Bulletin, No. 150, January, 1948, p. 77.

[2] ASTM Bulletin, No. 153, August, 1948, pp. 73 and 77.

A Special Subcommittee on Location of Exposure Test Sites, under the chairmanship of H. A. Nelson, has been cooperating with the A.S.T.M. Advisory Committee on Corrosion in considering various types of test sites for exposure tests. These include not only industrial locations but also marine exposures, tropical locations, and rural test sites.

NEW TENTATIVES

The committee recommends for publication as tentative the following seven new specifications and four new methods of test, as appended hereto:[3]

Specifications for:

Raw Castor Oil,
Dehydrated Castor Oil,
Heavy Mineral Spirits,
Secondary Butyl Acetate (85 to 88 per cent Grade),
Aluminum Pigment, Powder and Paste for Paints, to replace the Standard Specifications for Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266 - 41), and for Aluminum Pigment Paste for Paint (D 474 - 41),
Copper Phthalocyanine Blue,
Copper Powder, and

Methods of Test for:

Heat Bodying Rate of Drying Oils,
Abrasion Resistance of Organic Coatings by the Falling Sand Method,
Evaluating Degree of Resistance of Traffic Paint to Bleeding,
Para Red and Toluidine Red Pigments.

REVISIONS OF TENTATIVES

The committee recommends revisions of the following three tentative specifications and three tentative methods as indicated:

Tentative Specifications for:

Wood to Be Used as Panels in Weathering Tests of Paints and Varnishes (D 358 - 44 T), revise as appended hereto,[4]

Red and Brown Iron Oxide Pigments (D 84 - 46 T), revise as follows:

Section 2 (b).—Change to read as follows:

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to produce a paste or semipaste of satisfactory consistency. As received, it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	70
Nonvolatile matter in vehicle, min., per cent of vehicle	80
Moisture by distillation, max., per cent	2.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.5
Consistency by the Stormer viscosimeter:	
At shearing rate of 100 revolutions per 30 sec., min., g	700[a]
At shearing rate of 100 revolutions per 35 sec., max., g	1200[b]

[a] Equivalent to a rating of 125 Krebs' Units.
[b] Equivalent to a rating of 145 Krebs' Units.

Section 4 (*b*).—Add the following to this section which refers to the test for moisture: "except that the sample shall be weighed instead of measured and the results shall be expressed as percentage by weight instead of volume."

Venetian Red (D 767 - 44 T), revise by adding to Section 2 a new Paragraph (*b*) covering Paste in Oil to read the same as recommended above in Specifications D 84, relettering the present Paragraph (*b*) as (*c*).

Tentative Methods of Test for:

Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (D 283 - 47 T), revise by the addition of the following as a new Section 19:

19. *Coarse Particles Insoluble in Nitric Acid:* (*a*) *Procedure.*—Transfer the residue contained on a No. 200 (74-micron) sieve to a beaker, add 50 ml. of HNO_3 (1:3), and boil for 5 min. Cool and filter through a tared Gooch crucible, previously washed with

---

[3] These specifications and methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards Part II.

[4] These revised specifications and methods were accepted by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

HNO_3 (1:3) or a fritted-glass filter of appropriate porosity. Wash the insoluble residue on the filter with hot water. Dry the crucible or fritted-glass filter and contents at 105 to 110 C. and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles insoluble in nitric acid as follows:

$$\frac{A}{B} \times 100$$

where:

A = weight of residue, and

B = weight of original sample.

Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 47 T), revise as appended hereto,[4]

Specular Gloss of Paint Finishes (D 523 - 44 T), revise as appended hereto,[4]

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends the adoption as standard of the following two specifications and four methods, without revision:

Tentative Specifications for:

Black Synthetic Iron Oxide (D 769 - 44 T),

Pumice Pigment (D 867 - 46 T), and

Tentative Methods of:

Evaluating Degree of Resistance of Traffic Paint to Bleeding (D 868 - 46 T),

Test for Evaluating Degree of Settling of Traffic Paint (D 869 - 46 T),

Sampling and Testing Turpentine (D 233 - 44 T),

Testing Cellulose Acetate (D 871 - 46 T)

The committee also recommends the adoption as standard of the following five specifications and one method, with revisions as indicated:

Tentative Specifications for Titanium Dioxide Pigments (D 476 - 46 T), revise as appended hereto,[4] replacing the Standard Specifications for Titanium Dioxide Pigments (D 476 - 41).

Tentative Specifications for Raw Umber (D 763 - 44 T) and for Burnt Umber (D 764 - 44 T), combine into a single standard with a revision in Section 2 by addition of the following as a new Paragraph (*b*), relettering the present Paragraph (*b*) as (*c*):

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to produce a paste or semipaste of satisfactory consistency. As received, it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	55
Nonvolatile matter in vehicle, min., per cent of vehicle	80
Moisture by distillation,[a] max., per cent	2.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve) max., per cent of dry pigment	1.0

Tentative Specifications for Raw Sienna (D 765 - 44 T) and for Burnt Sienna (D 766 - 44 T), combine into a single standard with a revision in Section 2 making the same change as recommended above for Specifications D 763 - 44 T and D 764 - 44 T, except that the paste shall conform to the following requirements:

Pigment, min., per cent	60
Nonvolatile matter in vehicle, min., per cent of vehicle	80
Moisture by distillation, max., per cent	2.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve) max., per cent of dry pigment	1.0

Tentative Method of Test for Dry to No-Pick-Up Time of Traffic Paint (D 711 - 46 T), revise by the addition of the following note to Section 3 (*b*):

NOTE.—Progressive softening of the rubber by commonly used paint solvents may affect the end point. Adherent paint shall be removed from the wheel by wiping with a cloth moistened with acetone or methylethylketone.

Tentative Specifications for Orange Shellac and Other Lacs (D 237 - 43 T), revise as follows:

Section 1.—Change to read as follows:

1. These specifications cover three types and four grades of orange shellac and other

---

[a] Moisture by distillation shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95), except that the sample shall be weighedi nstead of measured and the results shall be expressed as percentage by weight instead of volume.

lacs as follows: type I, orange shellac, grades A, B, C, and D; type II, button lac and type III, garnet lac. Stick-lac and seed-lac are not included in these specifications.

Note 1.—In line 1 change "Grades A, B, and C" to read "Grades A, B, C, and D."

Note 2.—In line 1 delete "Grade E" and substitute "Type II," and in line 4 delete "Grade F" and substitute "Type III."

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—REQUIREMENTS FOR ORANGE SHELLAC AND OTHER LACS.

	Type I				Type II	Type III
	Grade A	Grade B	Grade C	Grade D	...	...
Matter insoluble in specified hot solvents, max., per cent	1.25	1.25	1.75	3.00	1.00	0.50
Iodine number, max.	...	...	...	24.5	18.0	18.0
Moisture, max., per cent	2.0	2.0	2.0	2.0	2.0	2.0
Wax, max., per cent	5.5	5.5	5.5	5.5	5.5	3.5
Orpiment, max., per cent	0.03	0.03	0.03	0.2	0.03	0.03
Matter soluble in water, max., per cent	0.5	0.5	0.5	0.5	0.5	0.5
Ash, max., per cent	1.0	1.0	1.0	1.0	0.5	0.5
Color	when specified, the color shall be no darker than that of a sample agreed upon by the seller and the purchaser.[a]					

[a] Attention is called to the fact that the seller and the purchaser must agree upon one of the two methods for determining color appearing in A.S.T.M. Methods D 29.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the following three standards be reverted to tentative and revised as appended hereto:[4]

Standard Specifications for:

C. P. Toluidine Toner (D 656 – 43),
C. P. Para Red Toner (D 475 – 41),

Standard Method of Test for:

Acetone Extract in Dry Lampblack and Dry Bone Black (D 305 – 31).

TENTATIVE REVISION OF STANDARD

The committee recommends a tentative revision of the Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 47) which comprises definitions of the following six terms: Japan, grinding japan, japan color, japan drier, stain, total luminous reflectance, luminous directional reflectance. These proposed definitions are appended hereto.[5]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in eight standards and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Specifications for Raw Tung Oil (D 12 – 41), revise Section 2 by changing the minimum limit for saponification number from "190" to "189."

Specifications for Raw Soybean Oil (D 124 – 41), revise as follows:

> *Section 1.*—Change to read: "these specifications cover raw soybean oil suitable for use in the paint and varnish industry."
>
> *Section 2.*—Change the requirement for saponification number from "190 min." to "189 to 195."
>
> Change the present minimum value of "0.924" for specific gravity at 15.5/15.5 C. to a range of "0.924 to 0.930."

Specifications for Perilla Oil, Raw or Refined (D 125 – 47), revise Section 2 by changing the requirement for maximum loss on heating at 105 to 110 C. from "0.2" to "0.3" per cent.

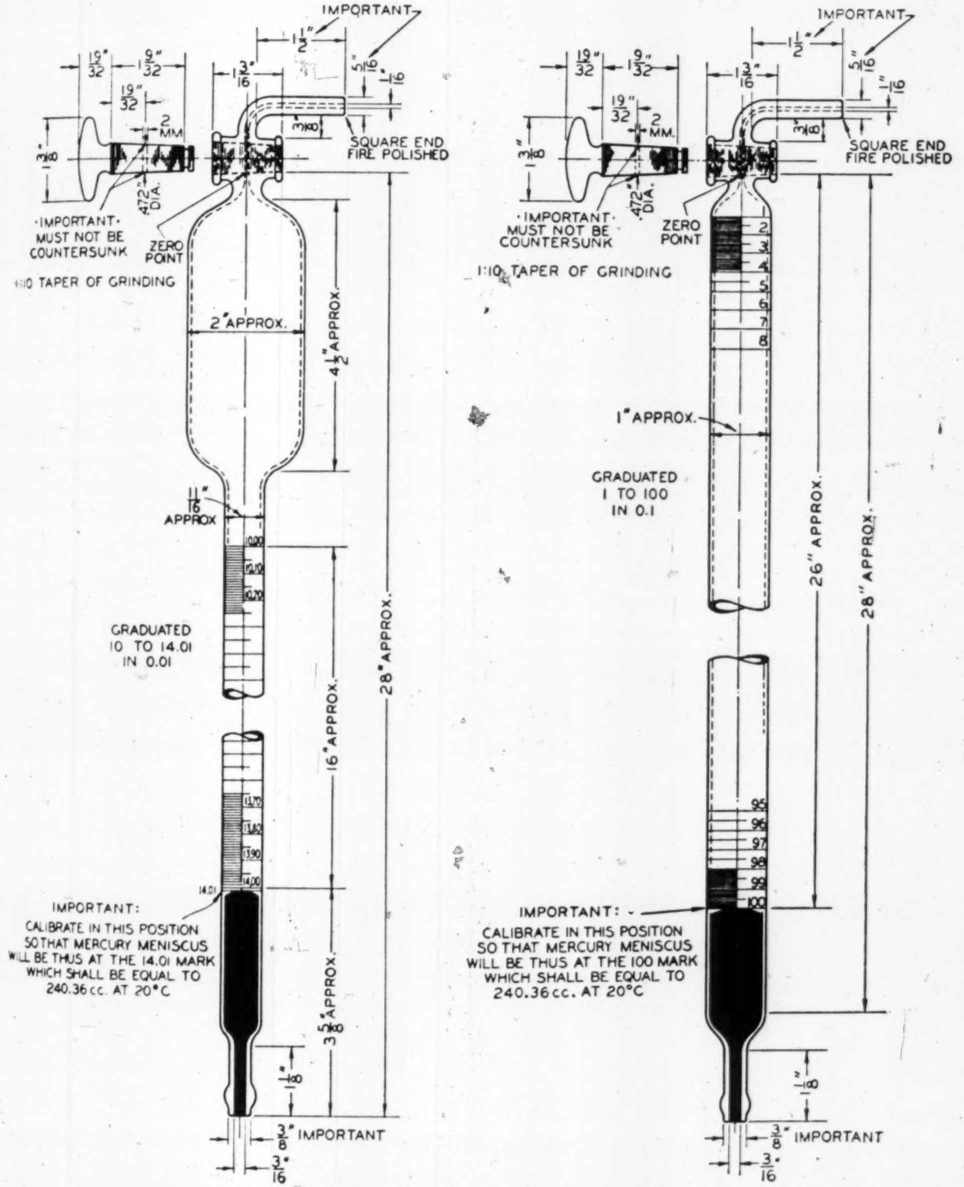
Specifications for Raw Linseed Oil (D 234 – 28), and for Boiled Linseed Oil (D 260 – 33), revise Section 2 by adding the following requirement for appearance "Clear and transparent at 65 C."; also change the requirement for maximum loss on heating at 105 to 110 C. from "0.2" to "0.3" per cent.

Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 46), revise Section 5, Item (*1*), which prescribes the thermometer for use in the distillation test, to read as follows:

> (*1*) The thermometer used shall be an A.S.T.M. Solvents Distillation Thermometer of suitable range conforming to the requirements for Thermometers 37 C., 38 C., 39 C., 40 C., 41 C., or 42 C. as prescribed in the

---

[5] These definitions were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.


IMPORTANT
1 1/2"
5/16"
1/16"
19/32"
1 9/32"
19/32"
2 MM
1 3/16"
1 3/8"
3/8"
SQUARE END FIRE POLISHED
·IMPORTANT· MUST NOT BE COUNTERSUNK
.472" DIA.
ZERO POINT
1:10 TAPER OF GRINDING
2" APPROX.
4 1/2" APPROX.
11/16" APPROX
10.00
10.10
10.20
GRADUATED 10 TO 14.01 IN 0.01
16" APPROX.
28" APPROX.
13.70
13.80
13.90
14.00
14.01
IMPORTANT: CALIBRATE IN THIS POSITION SO THAT MERCURY MENISCUS WILL BE THUS AT THE 14.01 MARK WHICH SHALL BE EQUAL TO 240.36 cc. AT 20°C
3 5/8" APPROX.
1 1/8"
3/8" IMPORTANT
3/16"
IMPORTANT
1 1/2"
5/16"
1/16"
19/32"
1 9/32"
19/32"
2 MM.
1 3/16"
1 3/8"
3/8"
SQUARE END FIRE POLISHED
·IMPORTANT· MUST NOT BE COUNTERSUNK
.472" DIA.
ZERO POINT
1:10 TAPER OF GRINDING
1
2
3
4
5
6
7
8
1" APPROX.
GRADUATED 1 TO 100 IN 0.1
26" APPROX.
28" APPROX.
95
96
97
98
99
100
IMPORTANT: CALIBRATE IN THIS POSITION SO THAT MERCURY MENISCUS WILL BE THUS AT THE 100 MARK WHICH SHALL BE EQUAL TO 240.36 cc. AT 20°C
1 1/8"
3/8" IMPORTANT
3/16"

(*a*) Nitrocellulose Tube. (*b*) Universal Tube.

FIG. 1.—Measuring Tubes for Nitrogen Determination.

Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).

Specifications and Tests for Soluble Nitrocellulose (D 301 - 33). The following changes are recommended after consideration of proposals received from the Technical Committee on Laboratory Apparatus of Committee E-1 on Methods of Testing:

> *Figure 2.*—In Fig. 2 (*b*) showing the universal measuring tube for the nitrogen determination, it is proposed to eliminate the calibration marks from the stopcock down to but not including the 1.0 deg. mark. Also due to the lack of uniformity of the glass tubing at this particular zone and to the fact that measurements are not ordinarily made in this region, this change is being made in the interest of avoiding the specifying of impractical laboratory glassware.
>
> In Fig. 2 (*a*) showing the nitrometer tube, the stopcock is being changed to conform with the construction now used in the nitrometer reaction bulb. This involves the use of uncolored glass, non-countersunk bores, and a countersunk channel for holding a retaining washer.
>
> These changes have been incorporated in the accompanying Fig. 1, which is recommended to replace the present Fig. 2 of Standard D 301.

Methods of Sampling and Testing Aluminum Powder and Paste (D 480 - 44), revise Section 4 (*b*), Item (*2*) by changing "27.0" to "24.5" dynes per cm.

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following three standard specifications which are being replaced by other specifications being submitted herewith, as mentioned earlier in this report:

Standard Specifications for:

Reduced Para Red (D 264 - 47),

Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266 - 41), and

Aluminum Pigment Paste for Paint (D 474 - 41).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman) prepared the new specifications for dehydrated castor oil and the test for heat bodying rate of drying oils presented herewith, and also recommended the revisions in the several specifications for drying oils presented in this report.

This subcommittee has under development tests for "break," acetone number, acetyl values, and diene value.

Subcommittee IV on Traffic Paint (W. G. Vannoy, chairman) prepared the new method for resistance of traffic paint to bleeding and recommended the adoption as standard of Methods D 711, D 868, and D 869.

Work is being continued by the subcommittee on the preparation of methods of test for night visibility, accelerated tests for traffic paint durability, and for degree of settling.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) recommended the adoption as standard of the Tentative Methods of Sampling and Testing Turpentine (D 233 - 44 T). A method for solvency of mineral spirits is under development by this subcommittee.

Subcommittee VI on Definitions (C. H. Rose, chairman) prepared the seven definitions of terms which are presented herewith as a tentative revision of Standard D 16. Definitions for the terms primer, sealer, filler, and size are in preparation.

Subcommittee VII on Accelerated Tests (H. A. Nelson, chairman) is completing a revision of Method D 609 which will provide an additional cleaning procedure for the preparation of steel panels used in testing paint.

The subcommittee is conducting work on humidity and immersion testing. Cooperative tests designed to establish the relationship between immersion tests made according to Method D 870, and humidity tests have been conducted. Both tests were made on two types of enamels at temperatures of 100, 110, and 120 F. Satisfactory correlation was obtained in the immersion tests, but the humidity results were too erratic for conclusions. Humidity testing will be studied further. The results of the immersion tests are to be presented in the form of a paper at the meeting of Committee D-1 in Detroit. The results indicate interesting conclusions as to the effects of temperature on immersion tests.

---

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

The subcommittee is conducting a test program on alternate immersion tests.

The program for studying the operation of light and water exposure apparatus is continuing in cooperation with the Cleveland Production Club. A report on the second cooperative test has been published.[7] The results indicate the importance of better control of the nature and uniformity of the water used, also, that the efforts to establish uniform temperature conditions have been a step in the right direction. A third cooperative test is being planned and, in the meantime, increased attention will be given to the possibilities of adopting deionized water as a means for eliminating the variations. This will require changes in the equipment which have already been made available by the manufacturers.

Subcommittee VIII on Methods of Chemical Analysis (C. Y. Hopkins, chairman) prepared the new tentative methods of testing para red and toluidine red pigments and recommended revisions in Methods D 480, D 305, and D 444 presented herewith. This subcommittee is studying changes in the methods covering the analysis of zinc dust pigment, chrome and blue pigments, ochres and umbers, and white pigments.

Subcommittee IX on Varnish (J. C. Weaver, chairman) has under way a cooperative program on a study of methods of test for drying time of varnish. Definitions and procedures for dust free, tack free to paper, tack free to metal, and print free time have been established for use in these tests.

Specifications and methods of test for phthalic anhydride are in preparation.

Revisions for the liquid drier specifications and methods are also under study. A test for nonvolatile content of varnish is being developed in which the sample is heated at 250 F. under a stream of carbon dioxide. A modification of the Run Kauri flexibility test, using pentaerythritol as a replacement for the Kauri resin, is being studied by cooperative tests.

A test for color of dried transparent films is in preparation. The subcommittee is undertaking a new project to compile, review and possibly publish conversion factors and data on some 40 viscometers now in use in the coatings industry.

Subcommittee X on Optical Properties (M. R. Paul, chairman) recommended as tentative the Proposed Revised Method of Test for Specular Gloss of Paint Finishes (D 523 – 44 T), as published in the ASTM Bulletin January, 1948, with a slight change in Section 3. The revised method is appended hereto.[1]

This subcommittee is continuing to work on new definitions for gloss and color; also methods of color specification, and procedures for measuring hiding power.

Subcommittee XI on Resins (J. J. Mattiello, chairman) has under way extensive programs covering both methods of test and specifications for various types of resins. In addition to the methods mentioned in last year's report, development work is under way on procedures for measurement of viscosity, solubility, refractive index and color resins. Other procedures being studied cover the determination of nitrogen in resins, free phenol in phenolic resins, and free chlorine in vinyl resins.

---

[7] "Weathering Machine Correlation in the Cleveland Club and Other Areas—Part I," *Official Digest*, Federation of Paint and Varnish Production Clubs, January, 1948.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) recommended the adoption as standard of Specifications D 237 with revisions as given previously in this report.

Subcommittee XV on Pigments (C. L. Crockett, chairman) has been very active in the development of new specifications for pigments and has also revised and brought up to date a number of the existing specifications. These various recommendations are covered earlier in the report.

Work is continuing on a revision of the Specifications for Pure Linseed Oil Putty for Glazing (D 317 – 33). Specifications for calcium carbonate are in preparation.

Subcommittee XVIII on Physical Properties (R. H. Sawyer, chairman) developed the falling sand abrasion test for paints presented herewith. Methods for measuring the thickness of films greater than 0.5 mils in thickness are being studied. Work is under way on methods for measuring adhesion, chalk resistance of pigments, specific gravity of pigments, and hardness of paint films. Studies are under way on the measurement of paste consistency, and a revision of Method 281 for oil absorption.

Subcommittee XXV on Cellulose Coatings and Related Materials (R. M. Carter, chairman) prepared the new specifications for secondary butyl acetate and for raw castor oil, presented as tentative herewith.

This subcommittee also recommended the revisions for immediate adoption in Standards D 268 and D 301 and the adoption of Tentative Method D 781.

In addition, a tentative revision of Standard D 301 was completed this year.

Subcommittee XXIX on Painting of Structural Iron and Steel (A. J. Eickhoff, chairman) is preparing a standard procedure for the field testing of paint coatings on structural steel.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 309 voting members; 171 members returned their ballots, of whom 157 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. H. ROSE,
Secretary.

REPORT OF COMMITTEE D-2*
ON
PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three meetings during the past year: in October, 1947, at Detroit, Mich., in February, 1948, at Washington, D. C., and in June, 1948, again at Detroit.

Committee D-2 has grown until its present organization embraces more than 600 individuals working on more than 100 subcommittees. Scheduling of meetings is becoming increasingly difficult and decreasingly satisfactory, particularly in connection with the Annual Meeting of the Society.

On October 1, 1947, T. G. Delbridge, "elder statesman" of petroleum technologists, and vice-chairman of Committee D-2 since 1930, retired from active duty and resigned as vice-chairman. L. C. Beard, Jr., was elected to fill the unexpired term of the vice-chairman.

Committee D-2 has suffered loss during the past year in the resignation of B. B. Bachman as chairman of Technical Committee A on Gasoline. Mr. Bachman's resignation was necessitated by heavy responsibilities in his company. Another loss was suffered when G. K. Brower, chairman of Technical Committee J on Aviation Gasoline, resigned from American Airlines to go with the John L. Ray Oil Co.

CHANGES IN COMMITTEE ORGANIZATION

Technical Committee K on Cutting Fluids.—Four subgroups have been organized under Section I on Laboratory Evaluation of Cutting Fluids (T. G. Roehner, chairman) on: (*1*) Analytical Tests (O. L. Brandes, chairman), (*2*) Physical Tests (E. W. Adams, chairman), (*3*) Cutting Ratio Tests (M. E. Merchant, chairman), and (*4*) Evaluation of Film Strength Testers of Cutting Fluids (E. M. Kipp, chairman).

Technical Committee L on Tractor Fuels (E. M. Hughes, chairman) has been organized with two sections: Section I on Classification and Nomenclature (Arthur B. Anderson, chairman, A. Schenkosky, vice-chairman, and J. R. Callaway, secretary), and Section II on Specifications (J. L. Nolan, chairman, B. G. Van Zee, vice-chairman, and H. P. Ferguson, secretary).

Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types.—Section G on Determination of Hydrogen has been organized under R. J. Matteson, chairman.

A Research Division on Corrosion Tests has been authorized under the chairmanship of F. D. Tuemmler.

A Research Division on Calorimetry has also been authorized.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Due to the fact that preprints of the 1947 Report of Committee D-2 were not available at the time of the 1947 Annual Meeting, only those recommendations which affected standards were reported to the Society at the June Meeting. Subsequent to the Annual Meeting, a supplementary preprint became available for letter ballot and all other recommendations in the 1947 Report[1] were referred to the Administrative Committee on Standards. They were accepted by the Standards Committee on September 4 and November 6, 1947, and are published in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

On April 7, 1948, the Administrative Committee on Standards accepted, on the recommendation of Committee D-2, a revision of the Tentative Specifications for Fuel Oils (D 396). The revised specifications under the A.S.T.M. Designation D 396 – 48 T have been published in pamphlet form.

ACTIVITIES OF TECHNICAL COMMITTEE AND SUBCOMMITTEES

Technical Committee A on Gasoline under its new chairman, Harold M. Smith, is carrying on the following activities:

Section I on Gum (C. A. Neusbaum, chairman) is working on a program designed to improve the accuracy and reproducibility of Methods D 381 – 46, Test for Existent Gum in Gasoline (Air-Jet Evaporation Method), D 525 – 46, Test for Oxidation Stability of Gasoline (Induction Period Method), and D 873 – 46 T, Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method).

Section V on Specifications and Significance of Tests (H. C. Dickinson, chairman) has written a statement on the significance of specifications as applied to gasoline which is being presented herewith[1a] for inclusion as an appendix to Specifications D 439.

Section VII on Tetraethyllead (C. M. Gambrill, chairman) has carried on an active program designed to improve Method D 526 – 42, Test for Tetraethyllead in Gasoline. The program resulted in a proposal for a revision of this standard with reversion to tentative, as appended hereto.[1a]

A small group on the determination of nonvolatile residue of jet fuels (C. A. Neusbaum, chairman) has been organized.

A new Section VIII has been organized on sludge and varnish-forming characteristics of motor gasoline, with S. S. Kurtz, Jr., as chairman.

Technical Committee B on Lubricating Oils (W. S. James, chairman) is studying the relationship between test data and service performance of lubricants in order to prepare test methods for the promotion of knowledge of lubricants. Several symposia have been held, the most recent being on Oil Procurement Practices, and Synthetic Lubricants.

A special committee (J. L. McCloud, chairman) has been appointed to study automotive crankcase oil classification systems.

Section U-III on Gear Oils (A. O. Willey, chairman) has two subgroups. Its Subgroup A on Automotive Gear Oils (H. C. Mougey, chairman) is endeavoring to rationalize differences in various specifications and classifications with regard to viscosity ranges; and its Subgroup B on Industrial Gear Oils (H. A. McConville, chairman) is preparing an oil classification, and is studying the important properties of gear oils. An informal symposium on experiences gained in this field was held in June, 1948.

Section U-IV on Hydraulic Power Transmission Liquids (H. R. Wolf, chairman) is working toward a classification, particularly regarding viscosity, to limit the number of required grades.

Section U-V on Instrument Oils (E. H.

---

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 271 (1947).

[1a] These revised specifications were accepted by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

Erck, chairman) is studying low-viscosity oils, both petroleum and synthetic, for use in the lubrication of light equipment. Wetting action, rust protective quality, oxidation stability and other properties of a fundamental nature are receiving attention.

Section U-VI on Air Compressor and Compressed Air Tools (C. M. Larson, chairman) is cooperating with the Compressed Air Institute in development of a classification for compressors and is planning tests in different types of compressors on various oils to determine limiting characteristics of lubricants for such units.

Section Q-II on Oxidation and Bearing Corrosion (H. R. Wolf, chairman) is working on a program aimed to devise test methods of small size for predicting the performance of oils and bearings.

Section Q-IV on Viscosity Stability (William Van Horne, chairman) is studying the effect of shear rate on viscosity stability of oils.

Technical Committee C on Turbine Oils (F. C. Linn, chairman) held an informal Symposium on the Care of Inhibited Turbine Oils and Turbine Lubrication[2] on February 9, 1948, in Washington, D. C. The following four papers were presented to an audience numbering approximately one hundred and fifty:

"Steam Turbines and Their Lubrication," by C. D. Wilson, Allis-Chalmers Manufacturing Co.

"The Care of the Lubricant and Maintenance of the Lubricating System for Central Station Turbine Equipment," by E. F. Walsh, Narragansett Electric Co.

"Service Experience with Turbine Oils," by C. L. Pope and O. V. Sprague, Eastman Kodak Co.

"The Use of Inhibited Oils in Central Station Equipment," by M. D. Baker, West Penn Power Co.

Section I on Oil Systems of Turbines (F. E. Rosenstiehl, chairman) is studying the preparation of turbine lubricating systems for service testing.

Section II on Rusting (W. P. Kuebler, chairman) is continuing the study of Method D 665 - 47 T, Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water, with a view to improving the test.

Section III on Oxidation (E. A. Reehl, chairman) has a program of study under way investigating factors that influence the oxidation stability of turbine oils.

Section IV on Emulsion Tests (M. D. Baker, chairman) having established through cooperative tests the precision of an Interfacial Tension Test of Oil Against Water by the Ring Method, Technical Committee C has proposed this method for publication as tentative.[3]

Technical Committee E on Burner Fuel Oils (M. A. Powers, chairman) prepared the revision of Tentative Specifications for Fuel Oils (D 396) which has been approved by Committee D-2 and published by the Society as D 396 - 48 T.

Technical Committee F on Diesel Fuels (C. G. A. Rosen, chairman) has prepared specifications for diesel fuel that are compatible with revised Tentative Specifications D 396.

Technical Committee G on Lubricating Greases (F. L. Wright, chairman) has carried on an active program of lubricating grease testing. As a result, the committee has formulated the following proposals:

(*1*) Publication as information of a Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease, as Appendix I.[4]

(*2*) Publication as tentative of the Proposed Method of Test for Evaporation Loss of Lubricating Greases and Oils, published as Appendix II to the 1947 Report of Committee D-2,[5] with minor revisions.

---

[2] ASTM BULLETIN, No. 152, May, 1948, p. 59 (TP 111).

[3] These methods and specifications were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[4] See p. 303.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 297 (1947).

(*3*) Adoption as standard of the Tentative Method of Test for Cone Penetration of Lubricating Greases (D 217 - 47 T) with a revised Fig. 4 as shown in the accompanying Fig. 1.

Technical Committee II on Light Hydrocarbons (W. G. Lovell, chairman) has through its sections carried out an active program of work.

Section II on Natural Gasoline (R. C. Alden, chairman) has conducted comprehensive tests which have resulted in a proposal by Technical Committee H, approved by Technical Committee J on Aviation Fuels, that certain modifications be made to Methods D 323 - 43, Test for Vapor Pressure of Petroleum Products (Reid Method) which will improve the reproducibility from "0.5" to "0.1" lb.

Section III on Chemical Utilization (R. F. Dunbrook, chairman) has proposed and Technical Committee H has approved the publication as information of the following three methods which appear as Appendices II, III, and IV,[6] respectively:

Proposed Methods of Test for:

Separation of Residue from Butadiene.
Butadiene-Dimer in Polymerization Grade Butadiene.
Determination of Nonvolatile Residue in Polymerization Grade Butadiene.

Also upon the recommendation of Section III, Technical Committee H has proposed for publication as tentative the Proposed Method for Determination of Butadiene Content of Polymerization Grade Butadiene by Koppers-Hinckley Method, published as Appendix III of the 1947 Report of Committee D-2.[7]

Section VI on Liquefied Petroleum Gas (G. R. Benz, chairman) has considered the definition of liquefied petroleum gas prepared by Committee D-3 on Gaseous Fuels. This definition was referred to Subcommittee XX on Nomenclature, of Committee D-2, for consideration. Section VI has also developed the Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases which Technical Committee H has proposed for publication as information as they appear in Appendix V.[8] This Section is also studying the Proposed Method of Sampling Liquefied Petroleum Gas in cooperation with Committee D-3.

Technical Committee J on Aviation Fuels (J. T. Hendren, chairman) has developed specifications for 115/145 grade aviation gasoline and proposed them as a revision of Specifications for Aviation Gasoline (D 910 - 47 T).

Technical Committee K on Cutting Fluids (O. L. Maag, chairman) has been quietly getting under way on a constructive program A Symposium was held at the January, 1947, meeting, the papers of which have been published in the ASTM BULLETIN, March, 1948. Eleven cooperating companies have agreed to set up lathes for running accelerated service tests. Data from the tests will be examined by Section II on Plant Evaluation of Cutting Fluids (M. E. McKinney, chairman).

Section III on Nomenclature, Definitions and Classifications (O. W. Boston, chairman) has completed a list of terms and definitions which is under consideration by the Technical Committee.

Technical Committee L on Tractor Fuels (Earl M. Hughes, chairman) held its organization meeting on January 9, 1948, in Chicago, Ill. The meeting was preceded by a Forum on Tractor Fuels at which the following papers were presented:[8a]

"Tractor Statistics as Related to the Farmer," by H. E. Everett, Implement and Tractor (an implement trade magazine).

"The Farmers' Interest in Tractor Fuels," by Prof. E. L. Barger, Iowa State College.

"A Review of Tractor Requirements as Related to Fuels," by Raymon Bowers, International Harvester Co.

---

[6] See pp. 309 to 317.

[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 300 (1947)

[8] See p. 318.

[8a] Issued as a separate publication, September, 1948

"The Complexity of Tractor Fuels and Taxation," by A. B. Anderson, Department of Taxation, State of Minnesota.

"The Effect of State Tax Regulations and State Specifications on Tractor Fuels from the Petroleum Industry Standpoint," by C. N. Hinkle, Standard Oil Co. (Indiana).

Division on Combustion Characteristics (D. B. Brooks, chairman) has carried out a program of setting up National Exchange Groups for maintaining accuracy of the detonation tests. An arrangement has been made between the Society and the National Bureau of Standards for the testing and certification of ASTM Reference Fuels. The sections of the Division have carried out the following activities:

Section I on Exchange Testing (Gustav Wade, chairman) has prepared test forms, log sheets, and framework charts to attain uniformity in reporting engine rating data on fuels by the several methods under the jurisdiction of the Division.

Section II on Precision (J. M. Campbell, chairman) prepared an analysis of the precision of test methods, which the Division has proposed to Committee D-2 for consideration in connection with all test methods. This analysis was referred to the Subcommittee on Coordination of Test Methods (F. D. Tuemmler, chairman) for report. The section is also preparing a statement regarding precision and reproducibility in conformance with statistical principles for inclusion as a part of the test methods for evaluating combustion characteristics of gasolines and diesel fuels.

Section III on Instrumentation (J. E. Taylor, chairman) is studying instruments that can be used alternatively or to replace the present bouncing pin. The Proposed Detonation Meter for Motor and Research Methods, under consideration by this Section, is published as information in Appendix VI.[9]

Section IV on Test Equipment (B. R. Siegel, chairman) is studying the redesign of the induction system.

Section V on Reference Materials (F. C. Burk, chairman) has been active in the formulation of plans for the testing and certification of reference materials.

Section VI on Operation and Maintenance (C. E. Habermann, chairman) has undertaken a study of the difficulties entailed in using *iso*octane and normal heptane as reference fuels.

Section VII on Editorial Revision (A. E. Becker, chairman) has been actively engaged in the editing of the ASTM Manual of Engine Test Methods for Rating Fuels which is now available and may be procured from Society Headquarters.

Subcommittee I on Pharmaceutical Tests (G. M. McNulty, chairman) has continued its study of the penetration characteristics of petrolatum.

Subcommittee III on Paraffin Wax (J. B. Rather, Jr., chairman) is undertaking a cooperative program to determine the merit of a new method for determining the oil content of both paraffin and microcrystalline wax. With respect to paraffin wax, the new procedure is being considered as a replacement for Method D 721 – 47. No method as yet has been standardized for microcrystalline waxes, but it is hoped that it will be possible to adopt the newly developed technique for that purpose.

Subcommittee V on Viscosity (J. C. Geniesse, chairman) is studying the determination of viscosity at high rates of shear and is following the viscosity studies being carried on in the American Petroleum Institute and The American Society of Mechanical Engineers. Subcommittee V has found that the calibration procedures for viscosimeters used on low-viscosity oils needs some improvement, hence, Method D 445 – 46 T, Test for Kinematic Viscosity, should be continued as tentative.

---

[9] See p. 328.

Subcommittee VI on Color (H. M. Hancock, chairman) has conducted cooperative tests on the Photoelectric Colorimeter Method published as information in the 1947 Report of Committee D-2.[10] It has proposed that Method D 155 – 45 T, Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter, be continued as tentative. Since the Army has indicated that the Emergency Method ES-32, Color of U. S. Motor Fuel (All Purpose) by Means of A.S.T.M. Color Standard is still useful, and since the Society has recommended the discontinuance of emergency methods, Subcommittee VI has proposed that ES-32 be published as tentative.

Subcommittee VII on Sulfur (H. M. Hancock, chairman) has cooperated with Section A on Determination of Chlorine and Sulfur by the Bomb Method (C. A. Neusbaum, chairman) of Subcommittee XI in attempting to reconcile Method D 129 – 44, Test for Sulfur in Petroleum Oils by Bomb Method with Method D 894, Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method, to produce a method applicable to all petroleum products to which the lamp sulfur method cannot be applied. Minor difficulties were encountered so that this reconciliation has not yet been accomplished. The subcommittee is cooperating with Section VI on Liquefied Petroleum Gas of *Technical Committee II* in the development of a sulfur method applicable to liquefied petroleum gas. The subcommittee has proposed that a Method for Determination of Sulfur in Petroleum Oils by the Quartz Tube Combustion Method be published as information in Appendix VII.[11]

Subcommittee VIII on Distillation (E. W. Dean, chairman) has continued through its Section A on Vacuum Distillation (H. M. Smith, chairman), the work toward the development of a vacuum distillation method.

Subcommittee X on Insoluble Content of Used Lubricating Oils (L. L. Davis, chairman) has proposed that the procedure for determination of sulfated residue in Method D 810 – 47 T, Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils be included also in Method D 893 – 46 T, Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils.

Subcommittee XI on Inorganic Elements in Lubricants (C. M. Gambrill, chairman) has an active program on the determination of sodium and potassium in lubricants. Flame photometric, polarographic and chemical methods are being investigated. This subcommittee has reported that cooperative testing of Method D 808 – 44 T, Test for Chlorine in Lubricating Oils by Bomb Method, and Method D 809 – 44 T, Test for Chemical Analysis for Phosphorus in Lubricating Oils, is not yet completed, hence these methods should continue as tentatives. The subcommittee has found that Methods D 810 – 47 T, Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils and D 811 – 44 T, Test for Chemical Analysis for Metals in Lubricating Oils have proved satisfactory in use and proposes that these tentatives be adopted as standard.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has conducted cooperative tests which have led to the proposal that the Proposed Method of Test for Acid and Base Numbers of Petroleum Oils by Alpha Naphtholbenzein Color Indicator published as information in Appendix VIII to the 1947 Report of

---

[10] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 307 (1947).

[11] See p. 332.

Committee D-2[12] be published as tentative with the title, Method of Test for Neutralization Value (Acid and Base Numbers) by Color Indicator Method. The subcommittee also proposes that the title of Method D 664 – 46 T be changed to Neutralization Value (Acid and Base Numbers) by Electrometric Titration. The publication of the Alpha Naphtholbenzein Method makes Method D 663 – 46 T, Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration, obsolete; consequently the subcommittee proposes that Method D 663 be withdrawn.

Subcommittee XIV on Water and Sediment (C. F. Ramey, chairman) has recommended that Method D 473 – 46 T, Test for Sediment in Fuel Oil by Extraction, be adopted as standard, after editorial correction.

Subcommittee XV on Measurement and Sampling (L. C. Burroughs, chairman) has pursued an active program of development of sampling procedures, temperature measurement, gaging procedures and volume correction tables. The chairman, together with J. G. Detwiler, has journeyed to England to confer with The Institute of Petroleum on collaboration and cooperation in the development of these tables.

Subcommittee XVI on Cloud and Pour Test (L. C. Beard, Jr., chairman) has continued the study of winter field tests on pour point stability.

Subcommittee XX on Nomenclature (S. S. Kurtz, Jr., chairman) has proposed the addition of a definition for Precipitation Naphtha to Standard D 288 – 47, Definitions of Terms Relating to Petroleum.

Subcommittee XXI on Crude Petroleum (A. J. Kraemer, chairman) has been exploring the desirability of further work on crude petroleum.

Subcommittee XXIII on Carbon Residue (F. I. L. Lawrence, chairman) has initiated a program to improve the correlation of the Conradson and Ramsbottom methods, particularly with reference to current products.

Subcommittee XXIV on Petroleum Sulfonates (G. M. McNulty, chairman) is studying the determination of inorganic salts in petroleum sulfonates and is also studying the determination of barium and calcium sulfonic salts.

Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types (S. S. Kurtz, Jr., chairman) has completed a cooperative test program on five diesel fuels for the Bureau of Ships. The analysis of jet propulsion fuels and of gasoline rich in olefins is being investigated. The determination of olefins and aromatics in distillate fuels is being actively pursued. A program is under way on the determination of olefinic unsaturation by chemical means. Extension of the scope of Method D 936 – 47 T, Test for Aromatic Hydrocarbons in Mixtures with Naphthenes and Paraffins by Silica Gel Adsorption, to gasoline with relatively low aromatic contents and to kerosine is being studied. The precision of Method D 940 – 47 T, Test for Determination of Purity by Measurement of Freezing Points, has been confirmed by cooperative testing. Several spectroscopic methods for analysis of petroleum products are being studied and a glossary of spectroscopic terms is being developed. The determination of hydrogen in petroleum products is being studied.

The Subcommittee on Coordination of Test Methods (F. D. Tuemmler, chairman) has continued to furnish editorial service to the subcommittees and technical committees.

NEW TENTATIVES

Committee D-2 recommends that the following six methods be published as tentative:

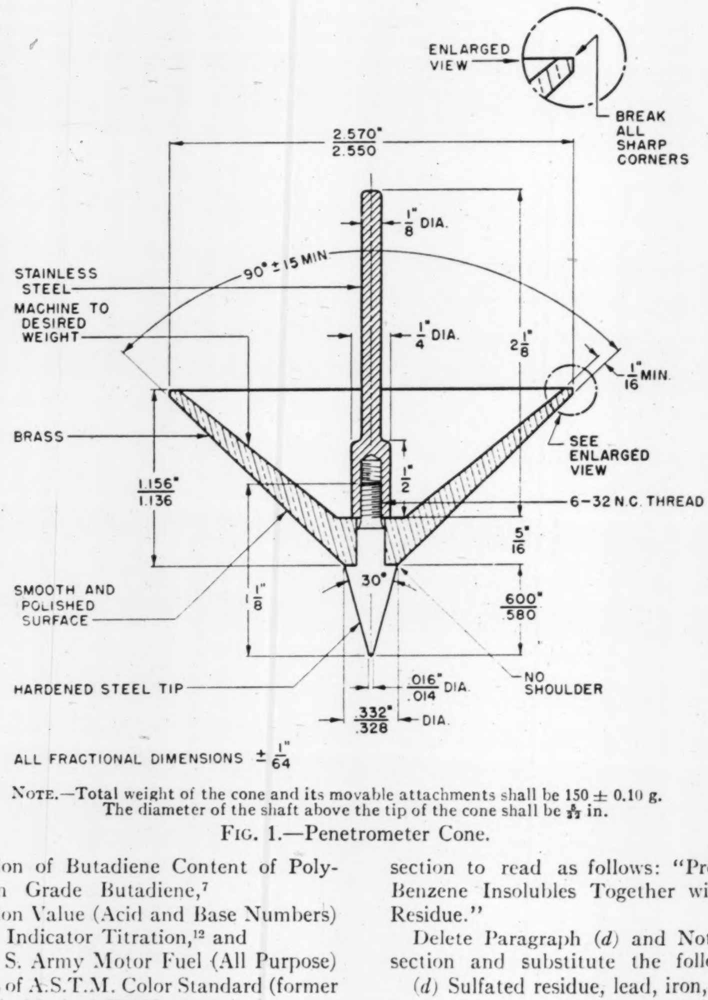
---

[12] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 331 (1947).

Tentative Methods of Test for:

Interfacial Tension of Oil Against Water by the Ring Method,[3]

Evaporation Loss of Lubricating Greases and Oils,[5]


ENLARGED VIEW
BREAK ALL SHARP CORNERS
2.570"
2.550
1" / 8 DIA.
90° ± 15 MIN.
STAINLESS STEEL
MACHINE TO DESIRED WEIGHT
1" / 4 DIA.
2 1/8"
1" / 16 MIN.
BRASS
SEE ENLARGED VIEW
1.156"
1.136
1" / 2
6-32 N.C. THREAD
5" / 16
30°
SMOOTH AND POLISHED SURFACE
1 1" / 8
.600"
.580
HARDENED STEEL TIP
.016"
.014 DIA.
NO SHOULDER
.332"
.328 DIA.
ALL FRACTIONAL DIMENSIONS ± 1" / 64

NOTE.—Total weight of the cone and its movable attachments shall be 150 ± 0.10 g. The diameter of the shaft above the tip of the cone shall be $\frac{3}{32}$ in.

FIG. 1.—Penetrometer Cone.

Determination of Butadiene Content of Polymerization Grade Butadiene,[7]

Neutralization Value (Acid and Base Numbers) by Color Indicator Titration,[12] and

Color of U. S. Army Motor Fuel (All Purpose) by Means of A.S.T.M. Color Standard (former Emergency Method ES-32).[13]

Tentative Specifications for:

Diesel Fuel Oils[3]

REVISION OF TENTATIVES

The committee recommends revision of the following three tentatives as indicated:

Tentative Methods of Test for:

Normal Pentane and Benzene Insolubles in Used Lubricating Oil (D 893 - 46 T), revised as follows:

Section 9.—Change the heading of this section to read as follows: "Procedure for Benzene Insolubles Together with Sulfated Residue."

Delete Paragraph (*d*) and Note 4 of this section and substitute the following:

(*d*) Sulfated residue, lead, iron, and copper in the benzene insolubles shall be determined in accordance with Sections 6 to 27 of A.S.T.M. Method D 810, Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils.

Tentative Specifications for:

Gasoline (D 439 - 40 T), revised by the addition of an appendix entitled "Significance of A.S.T.M. Specifications for Motor Gasoline," as appended hereto.[14]

---

[13] 1946 Book of A.S.T.M. Standards, Part III-A.

Aviation Gasolines (D 910 - 47 T), revised as appended hereto.[1a]

WITHDRAWAL OF TENTATIVE

In line with the proposal of Subcommittee XIII, the committee recommends that the Tentative Method of Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration (D 663 - 46 T) be withdrawn as obsolete.

TENTATIVE REVISION OF STANDARD

In line with the proposal of Technical Committee H, the committee recommends the following tentative revision to the Standard Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 43):

New Section.—Add the following as a new Section 23 to Method D 323:

23. *Modified Vapor Pressure Procedure for Aviation Gasoline.*—The modified vapor pressure procedure for aviation gasolines indicated in this section is required for aviation gasoline when high precision in the vapor pressure measurements is desired. The Reid vapor pressure method described in Sections 2 to 19 shall be used except for the following modifications:

(*a*) The ratio of the volume of the air chamber to the volume of the gasoline chamber shall be between the limits of 3.95 and 4.05 (Section 2(*e*)).

(*b*) The cooling bath shall be maintained at a temperature of 32 to 34 F. (Sections 4 and 14).

(*c*) The gage shall be checked at 7 lb. against a mercury column before each vapor pressure measurement to insure that it conforms to the requirements specified in Section 3(*b*). This preliminary check shall be made in addition to the final gage comparison specified in Section 18(*h*).

(*d*) The air chamber shall be adjusted to a temperature of 100 ± 0.3 F. before coupling to the liquid chamber (Section 17). This adjustment may be facilitated by rinsing the air chamber (Section 17(*a*)) with water at a temperature of approximately 102 F. and by the use of an air chamber bath for regulating the temperature of the air chamber with gage attached and air thermometer inserted (Section 17 (*c*)). However, any means of adjusting the air chamber temperature may be employed provided an air-thermometer reading constant at 100 ± 0.3 F. can be maintained for at least 5 min. just before coupling to the liquid chamber.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

In line with the proposal of Technical Committee A, the committee recommends that the Standard Method of Test for Tetraethyl Lead in Gasoline (D 526 - 42) be revised and that the revised method, appended hereto,[1a] be published as tentative.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Cone Penetration of Lubricating Grease (D 217 - 47 T), with corrected illustration of the penetrometer cone shown in the accompanying Fig. 1.

Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils (D 810 - 47 T),

Chemical Analysis for Metals in Lubricating Oils (D 811 - 44 T), and

Sediment in Fuel Oil by Extraction (D 473 - 46 T), with editorial revision as follows:

Section 1.—Change "90 per cent benzol" to read "industrial 90 benzene (A.S.T.M. Specifications D 837)."

REVISION OF STANDARD, IMMEDIATE ADOPTION

In line with the proposal of Subcommittee XX, the committee recommends the inclusion of the following definition in proper alphabetical order in the Standard Definitions of Terms Relating to Petroleum (D 288 - 47):

Precipitation Naphtha.—A naphtha suitable for determination of the precipitation number of lubricating oil (specification set forth in detail in A.S.T.M. Methods D 91, Test for Precipitation Number of Lubricating Oils, and A.S.T.M. Methods D 670, Test for Sludge Formation in Mineral Transformer Oil).

On the recommendation of the Division on Combustion Characteristics the committee recommends for immediate adoption the following revisions in the

Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 47):

(*1*) Inclusion as optional equipment of the Detonation Meter as described in Appendix VI.

(*2*) Substitution of the following for the present Section 15 on Reproducibility:

15. *Reproducibility of Results.*—Extensive data from independent laboratories over a number of years for many samples of conventional motor gasolines have shown a standard deviation of 0.5 octane number (an average deviation of about 0.4 octane number). Based on this standard deviation, the number of tests required to yield a rating of desired accuracy is given in the following table:

Accuracy Desired, Octane Number, plus or minus	Number of Tests Required to Obtain the Desired Accuracy		
	9 times out of 10	19 times out of 20	99 times out of 100
1.0	1	1	2
0.5	2	3	5
0.3	6	9	15

This table shows that a single rating can normally be expected to be within ±1.0 octane number of the true value in about 19 cases out of 20. On the other hand, if an accuracy of ±0.3 octane number is desired with a certainty of 90 per cent (9 times out of 10) it is necessary to obtain an average of six ratings in six different engines (preferably in six independent laboratories).

Although the above degree of reproducibility applies to conventional motor gasolines when careful attention is given to the details of test procedure and engine condition, it does not necessarily apply to fuels which differ materially from finished motor gasolines. In such cases, the reproducibility is likely to be lower and result in higher standard deviations.

EDITORIAL CHANGE IN TENTATIVE

The committee recommends that the Tentative Method of Test for Acid and Base Number of Petroleum Products by Electrometric Titration (D 664 - 46 T) be editorially changed by revising the title to read: "Tentative Method of Test for Neutralization Value (Acid and Base Numbers) by Electrometric Titration."

TENTATIVES CONTINUED AS TENTATIVE

The committee recommends that the following methods which have been published as tentative for two years or longer without revision be continued because revisions are under consideration or in prospect:

Tentative Methods of Test for:

Chlorine in Lubricating Oils by Bomb Method (D 808 - 44 T),

Chemical Analysis for Phosphorus in Lubricating Oils (D 809 - 44 T),

Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 - 45 T),

Foaming Characteristics of Crankcase Oils (D 892 - 46 T),

Kinematic Viscosity (D 445 - 46 T),

Oxidation Stability of Aviation Gasoline (Potential Gum Method) (D 873 - 46 T), and

Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 - 46 T), with editorial changes.

TABLE I.

Title	A.S.T.M. Designation	ASA Number
REVISION OF AMERICAN STANDARDS		
Test for Knock Characteristics of Motor Fuels by The Motor Method	D 357 - 47	Z11.37—1947
Analysis of Grease	D 128 - 47	Z11.16—1947
Test for Oil Content of Paraffin Wax	D 721 - 47	Z11.52—1947
Test for Cloud and Pour Points	D 97 - 47	Z11.5 —1947
Definitions of Terms Relating to Petroleum	D 288 - 47	Z11.28—1947
WITHDRAWAL OF AMERICAN STANDARDS		
Test for Water and Sediment in Petroleum Products by Means of Centrifuge	D 96 - 46	Z11.8 —1947
Test for Burning Quality of Kerosine	D 187 - 38	Z11.17—1939

AMERICAN STANDARDS

Table I shows the changes in status for the year ending June 1, 1948, of American Standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants, resulting from proposals made by Committee D-2.

Committee D-2 proposes that the Society recommend to Sectional Committee Z11 that Method D 526 – 42 (ASA No. Z11.48-1942), Test for Tetraethyllead in Gasoline, be discontinued as American Standard.

If the Society concurs with the recommendations of Committee D-2 listed in this report under Adoption of Tentatives as Standard and Revision of Standard, Immediate Adoption or such of these recommendations as are approved by the Society, the committee recommends that these standards be referred to Sectional Committee Z11 for approval as American Standards.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 95 voting members, the results of which will be reported at the Annual Meeting.[14]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. Dantsizen.
First Vice-Chairman, L. C. Beard, Jr.
Second Vice-Chairman, O. L. Maag.
Secretary, W. T. Gunn.

This report has been submitted to letter ballot of the committee, which consists of 95 voting members; 68 members returned their ballots, of whom 67 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. DANTSIZEN,
Chairman.

W. T. GUNN,
Secretary.

---

[14] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations which are covered in the Supplementary Report of Committee D-2:

Revision of Tentative Specifications for:

Gasoline (D 439 – 40 T), and
Diesel Fuel Oils (D 975 – 48 T).

Revision of Tentative Method of Test for:

Interfacial Tension of Oil Against Water by the Ring Method (D 971 – 48 T), Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 – 46 T),
Knock Characteristics of Motor Fuels by the Research Method (D 908 – 47 T),
Ignition Quality of Diesel Fuels by the Cetane Method (D 613 – 47 T),
Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 – 47 T), and
Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 – 47 T).

Revision and Reversion to Tentative of:

Standard Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 45).

Withdrawal of Standard Methods of:

Sampling Petroleum and Petroleum Products (D 270 – 33).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the new and revised tentatives appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

SUPPLEMENTARY REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS

At the meeting of Committee D-2 on Petroleum Products and Lubricants on June 23, 1948, the Technical Committees and Subcommittees of Committee D-2 presented numerous recommendations which indicated the desirability of filing this supplementary report with the Administrative Committee on Standards prior to publication of the 1948 A.S.T.M. Standards on Petroleum Products and Lubricants.

Division on Combustion Characteristics (D. B. Brooks, chairman) has proposed revisions of the detonation methods as follows:

(*a*) Revision of Method D 908, Test for Knock Characteristics of Motor Fuels by the Research Method to include:

(*1*) The Detonation Meter as optional equipment, and

(*2*) Revised Section on Reproducibility.

These revisions are identical with those in the Motor Method (D 357) as given in the main report[1] of Committee D-2.

The Division also submitted the Summary Report on Investigation of the Detonation Meter, Model 501, appearing in Appendix VIII which contains the results of the cooperative test program that furnished the basis for the recommendation that the detonation meter be adopted as optional equipment in the Motor and Research Methods.

(*b*) Revision of the Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 47 T) and the Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 - 47 T), comprising the substitution of the following for the present Section 13 on Reproducibility in each of these methods:

Reproducibility

13. The double scale used for reporting ratings by this method makes it difficult to apply the usual analysis for reproducibility. For those accustomed to using performance numbers, Tables XXIII[2] and XXIV,[2] reproducibility can be stated quite simply as follows:

Extensive data from independent laboratories over a number of years for many samples of conventional aviation gasolines have shown a standard deviation of 2.0 performance numbers (an average deviation of about 1.5 performance numbers). Based on this standard deviation, the number of tests required to yield a rating of desired accuracy is given in the following table:

Accuracy Desired, Performance Number Plus or minus	Number of Tests Required to Obtain the Desired Accuracy		
	9 times out of 10	19 times out of 20	99 times out of 100
4....	1	1	2
2....	3	4	7
1....	11	15	27

This table shows that it is normally necessary to obtain two ratings to be within plus or minus 4 performance numbers of the true value in about 99 cases out of 100. On the other hand, if an accuracy of plus of minus 1 performance number is desired with a certainty of 90 per cent (9 times out of 10) it is necessary to obtain an average of eleven ratings in eleven different engines (preferably in eleven independent laboratories). One performance number, Tables XXIII and XXIV, is equivalent to 0.5 octane number at the 90 octane level, to 0.3 octane number or 0.02 ml. of tetraethyllead per U. S. gallon of *iso*octane at the 100 octane number level, and to 0.07 ml. of tetraethyllead per gallon at the level of 1.25 ml. of tetraethyllead in *iso*octane.

---

[1] See p. 296.

[2] These tables will appear in the 1948 Appendix to the A.S.T.M. Manual of Engine Test Methods for Rating Fuels.

Although the above degree of reproducibility applies to conventional aviation gasolines when careful attention is given to the details of test procedure and engine condition, it does not necessarily apply to fuels which differ materially from finished aviation gasolines. In such cases the reproducibility is likely to be lower and result in higher standard deviations.

The tables showing conversion of octane numbers to performance numbers and conversion for Tetraethyllead in *iso*octane to performance numbers will appear as Tables XXIII and XXIV in the 1948 Appendix to the ASTM Manual of Engine Test Methods for Rating Fuels.

(*c*) Revision of the Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 - 47 T), comprising the substitution of the following for the present Section 13 on Reproducibility in this method:

Reproducibility

13. Extensive data from independent laboratories over a number of years for many samples of conventional Diesel fuels have shown a standard deviation of 1.4 cetane numbers (average deviation of about 1.0 cetane number). Based on this standard deviation, the number of tests required to yield a rating of desired accuracy is given in the following table:

Accuracy Desired. Cetane Number plus or minus	Number of Tests Required to Obtain the Desired Accuracy		
	9 times out of 10	19 times out of 20	99 times out of 100
2.....	1	2	3
1.....	5	8	13

This table shows that a single rating can normally be expected to be within plus or minus 2 cetane numbers of the true value in about 9 cases out of 10. On the other hand, if an accuracy of plus or minus 1 cetane number is desired with a certainty of 90 per cent (9 times out of 10), it is necessary to obtain an average of five ratings in five different engines (preferably in five independent laboratories.)

Although the above degree of reproducibility applies to conventional Diesel fuels when careful attention is given to the details of test procedure and engine condition, it does not necessarily apply to fuels which differ materially from finished Diesel fuels. In such cases the reproducibility is likely to be lower and result in higher standard deviations.

Technical Committee A on Gasoline (H. M. Smith, chairman) proposed a revision of Tentative Specifications for Gasoline (D 439 - 40 T) in February, 1948. The recommended revision comprised the lowering of the specified gum content of motor gasoline from "7 mg. per 100 ml." to read "5 mg. per 100 ml." This proposal was inadvertently omitted from the 1948 Report of Committee D-2, and is presented herewith.

Technical Committee C on Turbine Oils (F. C Linn, chairman) recommended that the Tentative Method of Test for Interfacial Tension of Oil Against Water by the Ring Method (D 971 - 48 T) which was accepted for publication as tentative in June, 1948, be revised as follows:

Section 3.—Add the following as a new Paragraph (*c*):

(*c*) Sample container, glass beaker or cylindrical vessel having a minimum diameter of 45 mm.

Section 4 (*a*).—In the last sentence, change the word "beaker" to read "sample container."

Section 7 (*a*).—Change to read as follows:

7. (*a*) Introduce 50 to 75 ml. of distilled water at a temperature of 25 ± 1 C. to a freshly cleaned sample container and place it on the adjustable platform of the tensiometer. Clean the platinum ring (Section 4 (*b*)) and suspend from the tensiometer. Raise adjusting platform until the ring is immersed in the water to a depth not exceeding 6 mm. in the center of the sample container as determined by visual observation.

Section 7 (*b*).—In the penultimate sentence, change the word "beaker" to read "sample container."

Technical Committee C also recommended the following editorial changes in Method D 157 - 36, Test for Steam Emulsion of Lubricating Oils:

Section 1.—Change to read as follows:

> 1. This method describes a procedure for the approximate evaluation of resistance of uninhibited turbine oils to emulsification. It is commonly used for turbine oils and may be used for other lubricating oils.

Add the following as a new Note 1, renumbering the subsequent notes accordingly:

> NOTE 1.—Results obtained on rust and oxidation inhibited turbine oils cannot be interpreted in the same manner as is the practice with uninhibited oils. Steam emulsion numbers of such inhibited oils are not reproducible and the existence of any correlation to emulsion in service is considered doubtful.

Technical Committee F on Diesel Fuels (C. G. A. Rosen, chairman) submitted a statement on "Significance of A.S.T.M. Specifications for Diesel Fuel Oils" to appear as an appendix to the Tentative Specifications for Diesel Fuel Oils (D 975 - 48 T), which were accepted for publication as tentative in June, 1948.

Technical Committee G on Lubricating Greases (R. C. Adams, chairman) recommends that the Tentative Method of Test for Evaporation Loss of Lubricating Greases and Oils (D 972 - 48 T) which was accepted as tentative in June, 1948, be revised as follows:

Section 2.—In the second sentence, change "23 hr." to read "22" hr.

Section 3.—Add the following as a new Paragraph (*e*):

> (*e*) *Flowrator.*—A rotameter calibrated to deliver air at a rate of 2.583 ± 0.02 g. per minute between 60 and 85 F. (2 liters per minute at standard temperature and pressure). It shall be furnished with a needle valve and mounted as shown in Fig. 1.

Section 4.—Delete this section, renumbering the subsequent sections accordingly.

Section 5 (b).—Change the third sentence to read as follows: "During this period, allow clean air to flow through the cell at the prescribed rate, 2.583 ± 0.02 g. per min. (2 liters per minute at standard temperature and pressure), as indicated by the flowrator."

In the same paragraph, delete the fourth sentence. In the last sentence, change "23 hr." to "22" hr. Delete the note at the end of this paragraph.

Section 5 (c).—In the first sentence, change "23 hr." to read "22 hr." Delete the second sentence.

Section 8.—Change to read as follows:

> 8. Results should not differ from the mean by more than the following amounts:

Repeatability Same Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
±2.5 per cent of mean	±10 per cent of mean

Supplement.—Substitute the following for footnote 3 of Section A1:

> The apparatus described in Section A1 is available from the Precision Scientific Co., Catalog No. 74929. The old cell formerly available under Catalog No. 74914 gives identical results but greater care must be exercised in handling the latter apparatus because of the less substantial construction.

Section A1 (a).—Change "tinned brass" to "stainless steel."

Section A1 (b).—Delete the last sentence in this paragraph.

Figure 1.—Substitute the accompanying Fig. 1 illustrating the evaporation test cell.[3]

Subcommittee XI on Determination of Inorganic Elements in Lubricants (C. M. Gambrill, chairman) recommended that the Tentative Method of Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 - 46 T) be revised as follows:

Section 1.—Change the last sentence to read: "The interference resulting from the presence of silicates in used oils and crudes is negligible."

---

[3] 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

Section 3 (b).—Change to read as follows: "(*b*) *Sample Cup*, platinum, 24 mm. in outside diameter at the bottom, 27 mm. in outside diameter at the top, 12 mm. in height outside, and weighing 10 to 11 g."

Section 3 (c).—Change to read as follows: "(*c*) *Firing Wire*, platinum, approximately No. 26 B. & S. gage."

Subcommittee XI also submitted the Proposed Method for Determination of Sodium in Lubricating Oils and Additives by Zinc Carbonate-Triple Acetate for publication as information as given in Appendix IX.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson Chairman) recommended that the Standard Method of Test for Saponification Number of Petroleum Products by Color Indicator Titration (D 94 – 45) be reverted to tentative and revised as follows:

Section 4 (a).—Change to read as follows:

4. (*a*) *Erlenmeyer Flask and Condenser.*—An Erlenmeyer flask, 300-ml. capacity, alkali resistant, to which is attached an adequate straight or mushroom-type reflux condenser. The straight-type condenser shall be fitted to the flask with a ground-glass joint; the mushroom-type condenser shall fit loosely to permit venting of the flask.

Section 9 (b).—Change to read as follows:

(*b*) The percentage of fatty oil or fat in a compounded petroleum product can be calculated from the saponification number of such a product if the actual saponification number of the fatty oil or fat is known. If the actual value is unknown but a sample of the added material is available, the saponification number of the additive is determined and used in calculating the fatty oil or fat content. If the actual saponification number is unknown and a sample of the added material is not available, the calculation for fatty oil content can be made with less accuracy, by using the average saponification number given in Table I for the fatty oil or fat known to be present. In any case, the reported value must be accompanied by a statement that the actual saponification number of the fatty oil was used in the calculation or that a stated, average value from Table I, was assumed to apply. The foregoing procedure is not applicable to calculation of fat content of compounded petroleum products containing easily saponifiable or acidic constituents, of unknown properties, other than the fatty oil or fat in question. However, if the saponification numbers of both the added fat in question and the compounded oil before blending with fat are known, the following formula is applicable:

$$\text{Fatty oil or fat, per cent} = \frac{(C - B)}{(F - B)} \times 100$$

where:

C = saponification number of the compounded oil containing added fat,

B = saponification number of the base oil before blending with fat, and

F = saponification number of the fatty oil or fat added.

TABLE I.—AVERAGE SAPONIFICATION NUMBERS OF FATTY OIL OR FAT COMMONLY USED IN COMPOUNDED PETROLEUM PRODUCTS.

Fatty Oil or Fat	Saponification Number	
	Average	Range
Castor	181	176 to 187
Coconut	256	251 to 261
Codliver	183	176 to 191
Cottonseed	194	187 to 197
Cottonseed, blown	218	210 to 225
Degras	[a]	85 to 150
Fish oil	[a]	100 to 200
Lard oil	195	190 to 198
Neatsfoot	197	193 to 204
Olive	193	189 to 197
Peanut	191	186 to 197
Rapeseed	175	170 to 179
Rapeseed, blown	205	195 to 216
Soybean	192	189 to 197
Sperm	128	120 to 140
Tallow, beef	197	194 to 200
Tallow, mutton	196	194 to 198

[a] The saponification numbers of degras and fish oil vary greatly according to their source and degree of refinement; therefore, it is not possible to give an average saponification number for these materials to be used for calculating the percentage in compounded oils.

NOTE 8.—If the blending material is an uncompounded refined oil stock, the saponification number of the stock (B) can be considered equal to zero in the above formula.

Subcommittee XV on Measurement and Sampling (L. C. Burroughs, chairman) proposed that the Standard Methods of

Sampling Petroleum and Petroleum Products (D 270 - 33) be withdrawn, as these methods are obsolete and this subcommittee expects to have a revised method ready for publication by April, 1949. Subcommittee XV also submitted for publication as information the Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products shown in Appendix X.

Subcommittee XXIV on Petroleum Sulfonates (G. M. McNulty, chairman) submitted for publication as information the Proposed Method for Analysis of Calcium and Barium Petroleum Sulfonates as shown in Appenxix XI.

Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types (S. S. Kurtz, Jr., chairman) recommended for publication as information the Proposed Method of Test for Benzene and Toluene by Means of Ultraviolet Spectrophotometry as shown in Appendix XII.

The recommendations appearing in this supplementary report have been submitted to letter ballot of the committee which consists of 95 voting members, the results of which will be reported to the Administrative Committee on Standards.[4]

Respectfully submitted on behalf of the committee,

C. DANTSIZEN,
Chairman.

W. T. GUNN,
Secretary.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

APPENDIX I

PROPOSED METHOD OF TEST FOR PERFORMANCE CHARACTERISTICS OF WHEEL BEARING GREASE[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method describes a procedure for obtaining data regarding the *probable* service behavior of wheel bearing greases. The evaluation includes consideration of the tendency of the grease, or of separated oil, to leak from the wheel hub in service and the tendency of the grease to form hard or varnish-like deposits upon the bearings or races.

(*b*) The method is not intended as the equivalent in all respects of a field test under service conditions.

Outline of Method

2. (*a*) The grease is distributed in a simulated front-wheel hub and spindle assembly. The assembly is run at a spindle speed of 440 ± 10 rpm. for 6 hr. with a temperature of 220 ± 2.5 F. at the spindle thermocouple. Leakage is measured and the condition of the grease and bearings is noted at the end of the test.

(*b*) If the results from a 6-hr. test are questionable, a 16-hr. test is made, imposing the same requirements on the sample.

Apparatus

3. The apparatus illustrated in Fig. 1 and described in detail in the Appendix is required. It consists of a simulated front-wheel hub and spindle assembly encased in an insulated cabinet. Means of measuring both ambient and spindle temperatures are provided.

Materials

4. *Naphtha, Precipitation,* conforming to the requirements in Section 4 of A.S.T.M. Method D 91, Test for Precipitation Number of Lubricating Oils.[2]

Procedure

5. (*a*) Weigh 90 ± 1 g. of sample on a flat plate. Pack by hand about 2 g. in the smaller bearing and 3 g. in the larger bearing. Distribute the balance of the weighed sample in a uniform layer on the inside of the hub (Note 1). Apply a very thin film of sample to the bearing races in the hub.

NOTE 1.—The 90-g. sample will fill the hub practically even with the bearing races and, with the exception of very fibrous greases, can be distributed readily and uniformly by use of a small spatula.

(*b*) Weigh separately the leakage collector and hub cap to the nearest 0.1 g. Place the leakage collector and the larger (inner) bearing in proper position on the spindle. Put the hub and small (outer) bearing on the spindle followed by the loose-fitting retainer ring. Tighten the castellated nut for holding the hub assembly in place until the hub ceases to coast when spun by hand; then slack off the nut approximately one-sixth of a turn until the hub rotates quite freely without end play. Lock the nut by inserting the cotter pin and screw on the hub cap. Put the V-belt in place, and close the cabinet.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1948.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

(*c*) Start the motor, and then turn on both heaters. Operate at a speed of 440 ± 10 rpm., while the temperature of the spindle is brought to 220 F. Maintain the temperature of the spindle at 220 ± 2.5 F., as indicated by the thermocouple inserted in it (Note 2).

NOTE 2.—This temperature will be attained with an ambient temperature near the hub of approximately 235 F. varying somewhat with room temperature, air currents, etc. This temperature should be reached in a maximum of 30 min., 20 min. generally being adequate. An ambient temperature of 235 F. will be maintained with the continuous heater on, and the intermittent heater cutting out at about 235 F., depending on room temperature.

(*d*) At the end of 6 hr. ± 5 min. from the time the motor was started, dismantle the apparatus while hot. Incline the hub assembly upon the bench, resting it upon the edge of the pulley and the small bearing end. Observe any change in consistency of the sample at

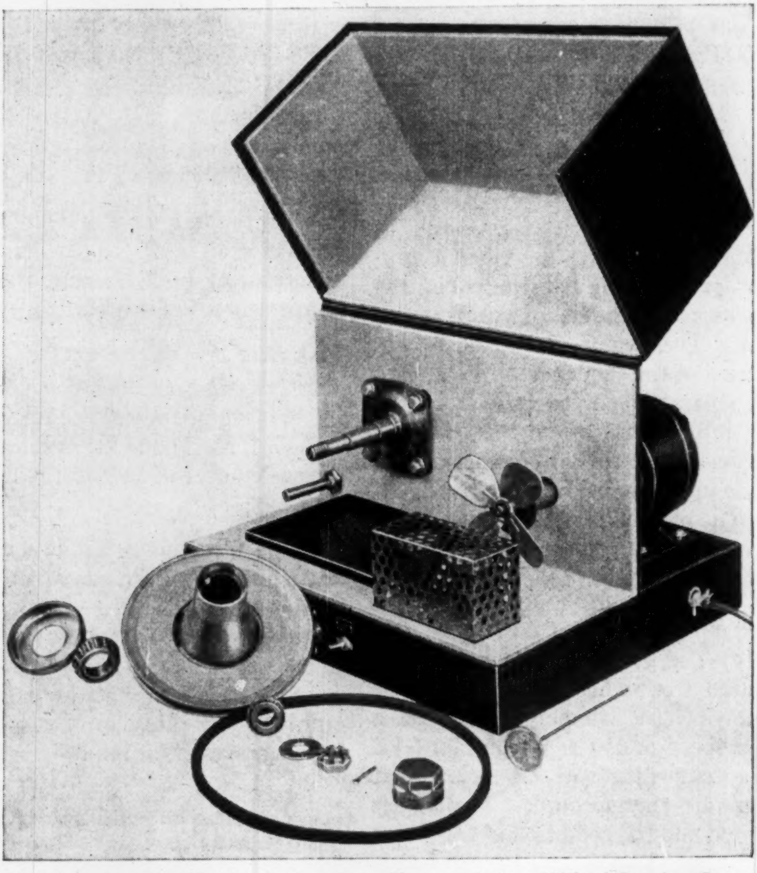


FIG. 1.—Apparatus for Testing Performance Characteristics of Wheel Bearing Grease.

the time of disassembly, and also after the grease has cooled to room temperature.

(*e*) Weigh separately the leakage collector and the hub cap to the nearest 0.1 g.

(*f*) Slush the two bearings in naphtha for 2 min. at room temperature to remove the grease film. Inspect for varnish, gum or lacquer-like deposits.

(*g*) *Long-Time Test.*—If the results of the 6-hr. test are inconclusive, repeat the procedure described in Paragraphs (*a*) to (*f*), inclusive, extending the time of test to 16 hr. ± 5 min.

Reporting

6. Report the following:

(*a*) Extent of change of the consistency of the grease, noting particularly whether the grease has become so thin that it runs from the inclined wheel as if it were a liquid, and also its condition after cooling to room temperature,

(*b*) Amount of leakage of grease or oil, or both, into the collector at the back of the hub and into the hub cap, and

(*c*) Presence of adherent deposit of varnish, gum, or lacquer-like material upon the bearing surface, which is evident after removal of the grease film. Naphtha insoluble soap residues, such as are encountered with sodium stearate greases, should not be confused with varnish or gums resulting from the deterioration of the lubricant.

Precision

7. Results on leakage measurements should not differ from the mean by more than the following amounts:

Repeatability One Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
±2g.	±4g.

APPENDIX

Apparatus

A1. *Main Spindle.*—The main spindle shall be constructed as shown in Fig. 2. The rear of the spindle shall be drilled to a depth of 1.5 in. for insertion of a thermocouple.

A2. *Bearing Hub.*—The bearing hub shall be constructed as shown in Fig. 3.

A3. *Hub and Spindle Assembly.*—The hub and spindle assembly shall consist of a simulated front-wheel and hub assembly mounted in a thermostatically controlled air bath. It shall be constructed as shown in Fig. 4. Two heaters shall be provided, one continuous and the other intermittent, mounted on the base of the apparatus, and controlled thermostatically. A fan shall be provided on the motor shaft to circulate air over the heater and hub.

A4. *Leakage Collector.*—A cup-shaped ring, used as a leakage collector, to catch any grease which may leak from the inner end of the hub (Note 3). This ring is removable and provides a method for determining the grease loss. It slips over the spindle and is held in place by the large bearing.

Note 3.—The regular grease retainer is not used because it is desirable in a laboratory test to accelerate any leakage which may occur. Also defective retainers are frequently found in actual service.

(See Figs. 2 to 4, pp. 306 to 308.)

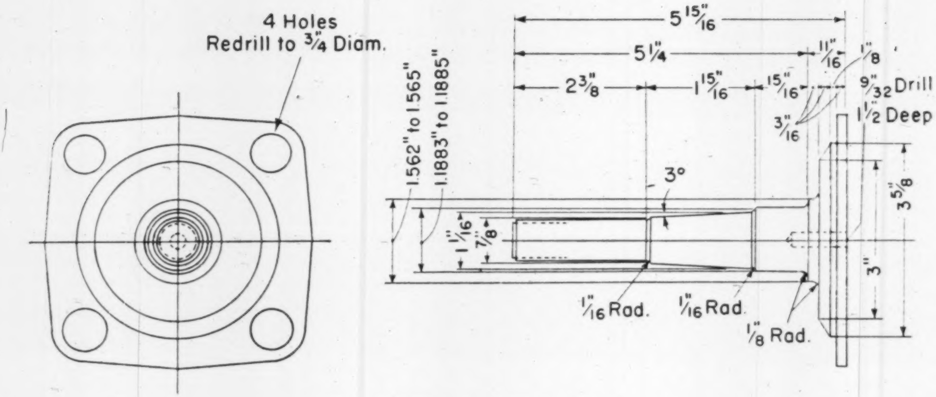

4 Holes
Redrill to 3/4 Diam.
1.562" to 1.565"
1.1883" to 1.1885"
5 15/16"
5 1/4
11/16"
1/8"
2 3/8"
1 15/16"
15/16"
9/32 Drill
1 1/2 Deep
3/16"
3°
1 1/16"
7/8"
3 5/8
3"
1/16 Rad.
1/16 Rad.
1/8 Rad.

FIG. 2.—Details of Main Spindle.

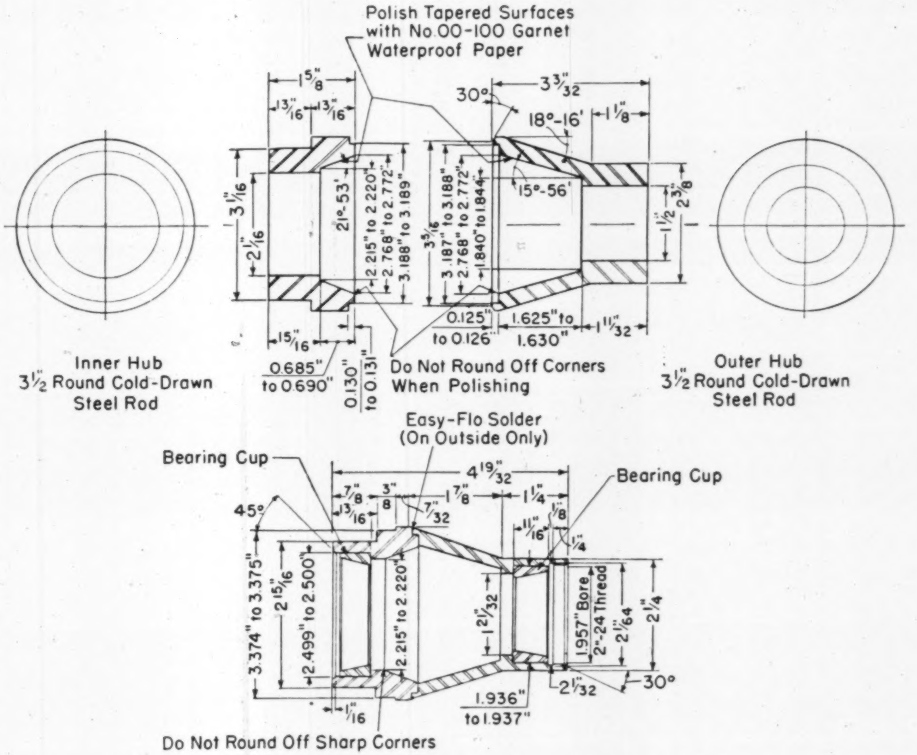
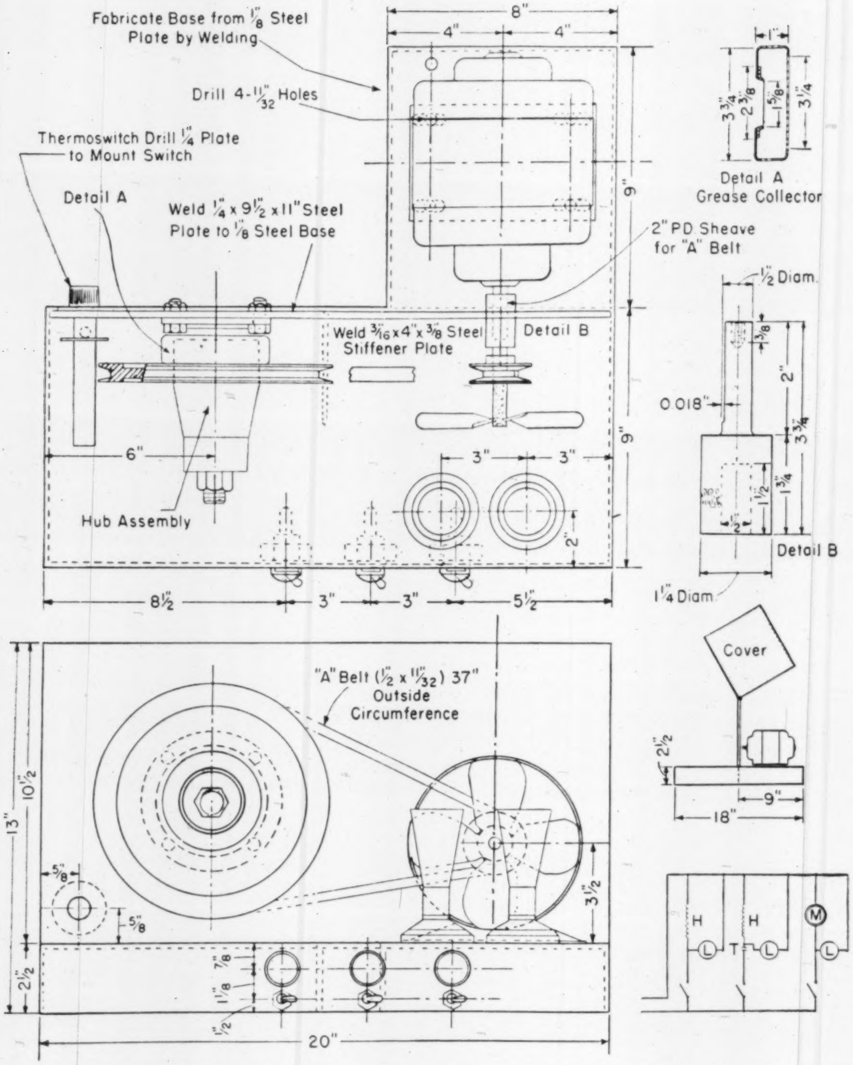

Polish Tapered Surfaces with No.00-100 Garnet Waterproof Paper
1⅝"
13/16"
13/16"
3 1/16
2 1/16
21°-53'
2.215" to 2.220"
2.768" to 2.772"
3.188" to 3.189"
15/16"
0.685" to 0.690"
0.130" to 0.131"
Do Not Round Off Corners When Polishing
Inner Hub
3½ Round Cold-Drawn Steel Rod
30°
3 3/32"
18°-16'
1⅛"
15°-56'
3 3/16
3.187" to 3.188"
2.768" to 2.772"
1.840" to 1.844"
2⅜"
1½"
0.125" to 0.126"
1.625" to 1.630"
1 11/32"
Outer Hub
3½" Round Cold-Drawn Steel Rod
Easy-Flo Solder (On Outside Only)
Bearing Cup
Bearing Cup
4 19/32"
7/8"
3/8"
1⅞"
1¼"
13/16"
7/32"
11/16"
1/8"
1/4"
45°
3.374" to 3.375"
2 15/16
2.499" to 2.500"
2.215" to 2.220"
1 21/32
1.957" Bore
2"-24 Thread
2 1/64
2¼"
2 1/32
30°
1.936" to 1.937"
1/16"
Do Not Round Off Sharp Corners

FIG. 3.—Details of Bearing Hub.


Fabricate Base from 1/8" Steel Plate by Welding
Drill 4-11/32" Holes
Thermoswitch Drill 1/4" Plate to Mount Switch
Detail A
Weld 1/4" x 9 1/2" x 11" Steel Plate to 1/8 Steel Base
Weld 3/16 x 4" x 3/8 Steel Stiffener Plate
Detail B
2" PD Sheave for "A" Belt
Hub Assembly
8"
4"
4"
9"
9"
6"
3"
3"
2"
8 1/2
3"
3"
5 1/2
1"
3 3/4"
2 3/8
1 5/8
3 1/4
Detail A
Grease Collector
1/2 Diam.
3/8
2"
0.018"
3 3/4
1 3/4
1 1/2
1/2
Detail B
1 1/4 Diam
Cover
2 1/2
9"
18"
"A" Belt (1/2 x 11/32) 37" Outside Circumference
13"
10 1/2
5/8"
5/8
3 1/2
2 1/2
7/8
1 1/8
1/2
20"
H
H
M
L
T
L
L

NOTE.—The spindle must be mounted to the case using fibre spacers next to the insulation on the inside of the case and also on the outside of the case. If spacers are not used a very high ambient temperature may be necessary to attain the spindle temperature of 220 ± 2.5 F.

FIG. 4.—Details of Hub and Spindle Assembly.

APPENDIX II

PROPOSED METHOD OF TEST FOR SEPARATION OF RESIDUE FROM BUTADIENE[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test describes a procedure for the separation of polymerization grade or recycle butadiene into a distillate consisting of butadiene together with C_5 and lighter hydrocarbons, and a residue containing butadiene dimer (4-vinyl-1-cyclohexene), non-volatile matter, and styrene if present.

NOTE 1.—If the butadiene sample is to be analyzed only for dimer or styrene, or both, so that it is unnecessary to collect the distillate, use the distillation procedure of the Proposed Method of Test for Butadiene Dimer in Polymerization-Grade Butadiene.[3]

Outline of Method

2. The sample is distilled through a small, simply designed fractionating column with rapid throughput and slight reflux (Fig. 1). The overhead vapors are condensed for use in analyses requiring samples free from dimer, styrene, and non-volatile matter. The residue is further purified for analysis for dimer or styrene, or both.

NOTE 2.—The separation is sufficiently quantitative so that the distillate may be used in analyses, such as those for butadiene and C_5 hydrocarbons, in which dimer, non-volatile matter, and styrene interfere. However, the residue must be further purified to remove traces of C_4 and C_5 hydrocarbons before its analysis for dimer or styrene, or both.

Apparatus

3. The apparatus required is illustrated in Fig. 1 and consists of the following parts:

(*a*) *Fractionating Column*, vacuum-jacketed and packed with $\frac{1}{8}$ in. glass helices, constructed as detailed in Fig. 1.

(*b*) *Distillation Flask*, flat-bottomed, 125-ml. capacity, with a 24/40 standard taper ground-glass joint.

(*c*) *Receiver*, as shown in Fig. 1.

(*d*) *Graduated Cylinder*, capacity of 50 ml., graduated in 0.5-ml. subdivisions, accurate to 0.24 ml.

(*e*) *Water Bath.*—Use a 1000-ml. beaker.

(*f*) *Dry Ice-Acetone Bath.*—Use a wide mouth, 1-qt. capacity, Dewar flask.

(*g*) *Chloroform Trap, Graduated.*—This trap may be constructed from a 15-ml. centrifuge tube by sealing to it a side tube and an inner tube which extends approximately to the 5-ml. mark. Provide a drying tube filled with calcium chloride at the opening to the air.

(*h*) *Thermometer*, range of −40 to 50 C., graduated in 1 C. intervals, mercury-filled.

Materials

4. The following reagents and materials are required:

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1948.

[2] This method was developed and has been cooperatively tested by the Butadiene Producer's Committee on Specifications and Methods of Analyses of the Office of Rubber Reserve. It appears in the Butadiene Laboratory Manual, Office of Rubber Reserve, as Method No. 2.1.56.2.

[3] See p. 312.

(*a*) *Acetone, c.p.*
(*b*) *Carbon Tetrachloride, c.p.*
(*c*) *Chloroform, c.p.*, alcohol-free.

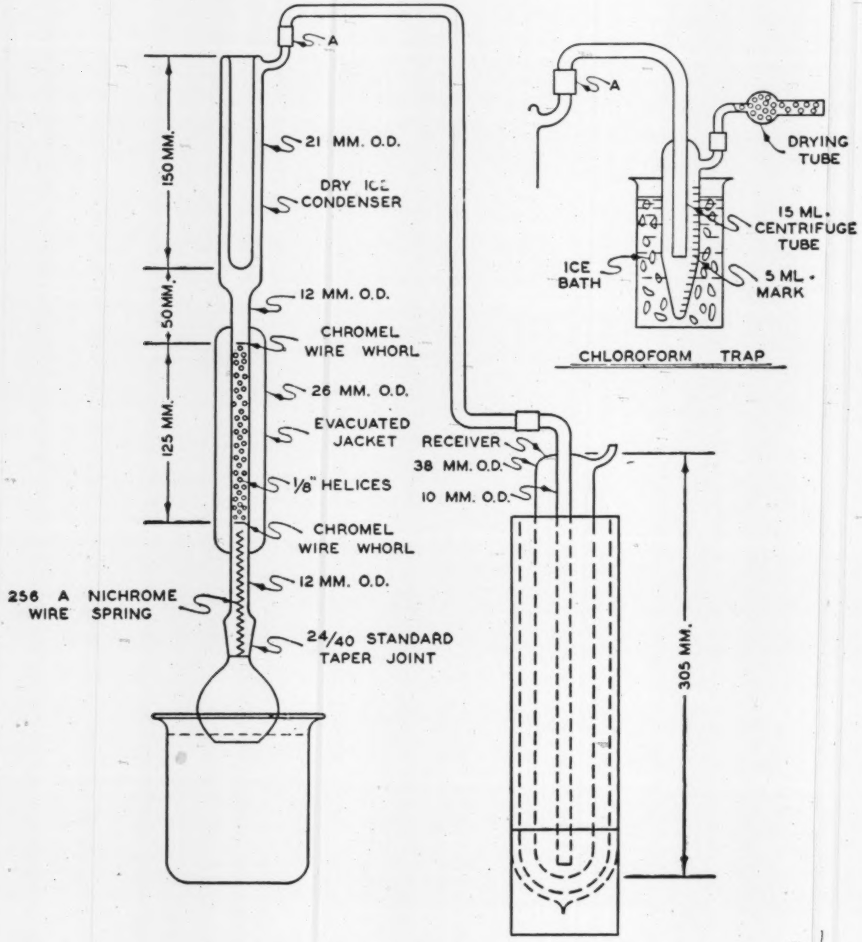

A
150 MM.
21 MM. O.D.
DRY ICE CONDENSER
50 MM.
12 MM. O.D.
CHROMEL WIRE WHORL
125 MM.
26 MM. O.D.
EVACUATED JACKET
1/8" HELICES
CHROMEL WIRE WHORL
12 MM. O.D.
256 A NICHROME WIRE SPRING
24/40 STANDARD TAPER JOINT
RECEIVER
38 MM. O.D.
10 MM. O.D.
305 MM.
A
DRYING TUBE
15 ML. CENTRIFUGE TUBE
ICE BATH
5 ML. MARK
CHLOROFORM TRAP

FIG. 1.—Assembly of Distillation Apparatus.

Preparation of Apparatus

5. Assemble the apparatus as shown in Fig. 1, mounting the column on a ring stand and maintaining it upright by means of a clamp around the condenser. Connect another clamp to the ring stand below the condenser for holding the 125-ml. flask containing the sample. Immerse the receiver in a dry ice-acetone mixture contained in a Dewar flask. Then connect the vapor outlet of the column to the tube sealed into the receiver using glass tubing and making glass-to-glass connections by means of neoprene tubing. After assembly, introduce pulverized dry ice into the column condenser to a height of 35 mm.

Procedure

6. (*a*) Cool the sample, a 50-ml. graduated cylinder, and the distillation flask to about −20 C. Introduce into flask, by means of the graduate, 50 ± 0.25 ml. of chilled sample whose temperature is known to the nearest 1 C. Connect the flask to the column and fasten the neck securely with the clamp provided.

(*b*) Introduce about 700 ml. of water at a temperature of 45 C. into the water bath and start the butadiene refluxing by raising the water bath to immerse the flask momentarily. Then for a period of 1 min. agitate the dry ice in the condenser and the flask momentarily at required intervals to maintain a reflux rate of about 2 drops per sec. and to condense all vapors. Then discontinue the agitation of the dry ice and immerse about 3 mm. of the flask to reduce the reflux rate to about 2 drops every 3 sec. and to start the distillation. Continue the distillation, adding more dry ice to the condenser if necessary and agitating the dry ice or applying constant pressure to it with a stirring rod or test tube if the reflux rate becomes too low.

(*c*) When all of the sample has been distilled from the flask, as indicated by a rise of condensate in the inlet arm of the receiver, remove the residual dry ice from the condenser and completely submerge the flask in the water bath for a period of 5 to 10 min. Then disconnect the receiver and mix the distillate thoroughly. Use this distillate for analyses in which dimer, non-volatile matter, and styrene interfere.

(*d*) Introduce 20 ml. of chloroform into the column through the vapor outlet and place a few pieces of dry ice in the condenser. Gradually heat the water to approximately 100 C. by passing steam into the water and allow the chloroform vapors to rise in the column until they start to reflux rapidly. Then remove any remaining dry ice from the condenser and attach the graduated trap, immersed in ice, to the vapor outlet, *A*. Continue heating until the vapors which gradually rise in the cooling chamber distill from the top of the column. To insure complete removal of butadiene, continue the distillation for about 3 min. until at least 6 ml. of chloroform have been collected in the trap.

(*e*) Pour 10 ml. of carbon tetrachloride into the column through the vapor outlet to remove any residue on the packing. Surround the flask with water at a temperature of 10 to 20 C. and allow the column to drain for a few minutes. Then disconnect the flask from the column and use the residue in analyses for dimer or styrene, or both.

APPENDIX III

PROPOSED METHOD OF TEST FOR BUTADIENE DIMER IN POLYMERIZATION GRADE BUTADIENE[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test describes a procedure for the determination of butadiene dimer (4-vinyl-1-cyclohexene) in polymerization grade butadiene. The interference of inhibitor (*p*-tertiary butyl catechol) and nonvolatile butadiene polymers is negligible. However, acetylene dimers or polymers, if present, may interfere.

NOTE 1.—The method includes a distillation procedure for isolating the dimer and non-volatile matter before analysis. If determinations of butadiene and C_5 hydrocarbons on a dimer-free sample are also desired, use the distillation procedure of the Proposed Method of Test for Separation of Residue from Butadiene.[3]

Outline of Method

2. The butadiene sample, with chloroform added as a chaser, is rapidly distilled through a small, simple distillation column (Fig. 1). The dimer is retained in the chloroform residue while the butadiene is completely expelled without being condensed. The residue is then analyzed for dimer content by the procedure for the determination of olefin described in A.S.T.M. Method D 875, Test for Olefins and Aromatics in Petroleum Distillates.[4]

Apparatus

3. The following apparatus is required:

(*a*) *Distillation Apparatus*, vacuum jacketed and packed with $\frac{1}{8}$ in. glass helices constructed as detailed in Fig. 1.

(*b*) *Distillation Flask*, flat-bottomed, 125-ml. capacity, with a 24/40 standard taper ground-glass joint.

(*c*) *Water Bath.*—Use a 1000-ml. beaker.

(*d*) *Bromination Flask.*—Use a 500-ml. glass-stoppered iodine-number flask.

(*e*) *Thermometer*, range of −40 to 50 C., graduated in 1 C. intervals, mercury-filled.

Materials

4. The following materials are required:

(*a*) *Carbon Tetrachloride, c.p.*

(*b*) *Chloroform, c.p.*, alcohol-free.

Preparation of Apparatus

5. Assemble the apparatus as shown in Fig. 1 mounting the column on a ring stand in a hood near a source of steam. Maintain the column upright by means of a clamp around the condenser and

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1948.

[2] This method was developed and has been cooperatively tested by the Butadiene Producer's Committee on Specifications and Methods of Analyses of the Office of Rubber Reserve. It appears in the Butadiene Laboratory Manual, Office of Rubber Reserve, as Method No. 2.1.15.

[3] See p. 309.

[4] 1946 Book of A.S.T.M. Standards, Part III-A.

connect a second clamp to the ring stand below the condenser for holding the 125-ml. flask. Fill about one half of the cooling chamber of the condenser with pulverized dry ice.

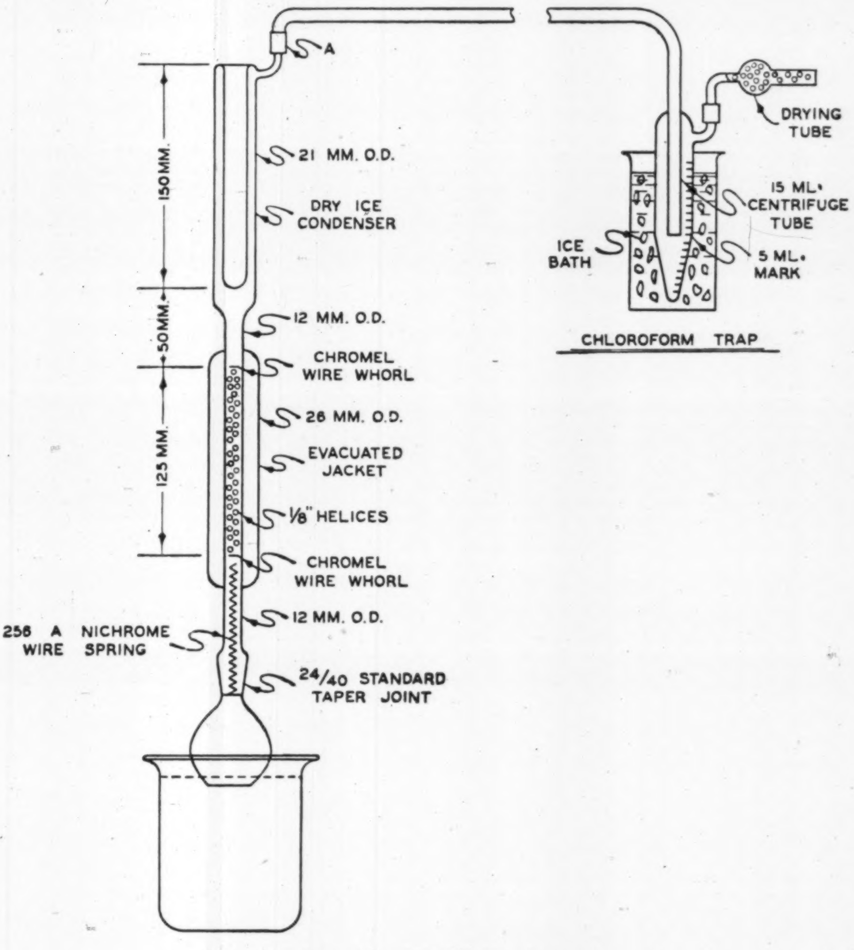

A
150 MM.
21 MM. O.D.
DRY ICE CONDENSER
50 MM.
12 MM. O.D.
CHROMEL WIRE WHORL
26 MM. O.D.
125 MM.
EVACUATED JACKET
1/8" HELICES
CHROMEL WIRE WHORL
12 MM. O.D.
256 A NICHROME WIRE SPRING
24/40 STANDARD TAPER JOINT
DRYING TUBE
15 ML. CENTRIFUGE TUBE
ICE BATH
5 ML. MARK
CHLOROFORM TRAP

FIG. 1.—Assembly of Distillation Apparatus.

Procedure for Distillation

6. (*a*) Introduce approximately 10 ml. of chloroform into the 125-ml. flask. Cool the flask and contents, a 50-ml. graduated cylinder, and the sample to approximately −20 C. Introduce into the flask by means of the graduated cylinder 50 ± 0.25 ml. of the sample whose temperature is known to the nearest 1 C. Connect the flask to the column and fasten the neck securely with the clamp provided. If desired, the clamp around the condenser may be loosened and the weight of the column and flask may be supported entirely by the clamp around the neck of the flask. This permits raising or lowering the assembly by manipulation of one clamp and makes it unnecessary to raise and lower the water bath.

(*b*) Half fill the water bath with water at 10 to 20 C. and start the butadiene boiling by immersing the flask in the bath. Condense all the vapors until the column is wetted completely. Then rapidly distill the butadiene off through the top of the column with a reflux rate of about 1 drop per sec. raising the temperature of the water by passing steam into it and by allowing the amount of dry ice in the condenser to decrease.

(*c*) When most of the butadiene has distilled, the bottom of column has become warm, and the temperature of bath has risen to about 61 C. (boiling point of chloroform), remove the dry ice from the condenser and attach the trap, immersed in ice, to the condenser outlet. Heat the water bath almost to its maximum temperature and allow the chloroform vapors to rise gradually in the condenser until they distill off through the top of the column. Then to insure complete removal of butadiene, continue the distillation for about 3 min. until at least 4 ml. of chloroform have collected in the trap. Then discontinue heating and remove the flask from the bath.

NOTE 2.—A normal distillation should require less than 30 min.

(*d*) Pour 10 ml. of carbon tetrachloride into the column through the vapor outlet to remove any residue on the packing. Again immerse the flask in water at 10 to 20 C. and allow the column to drain for a few minutes before disconnecting the flask.

Procedure for Bromination

7. Analyze the residue, prepared as described in Section 6, for dimer content by the bromination procedure for the determination of olefins as described in Method D 875, observing the requirements of the following Paragraphs (*a*) to (*c*):

(*a*) Use an amount of the residue such that not more than 12 ml. of the 0.5 *N* bromide-bromate solution are required for the titration. If all of the residue is used, transfer the chloroform solution directly to a 500-ml. iodine-number flask, rinse the distillation flask twice with approximately 5 ml. of carbon tetrachloride, and add the rinsings to the iodine-number flask. If only a portion of the residue is used, dilute a measured percentage of the chloroform solution of the residue with a measured amount of carbon tetrachloride and take an aliquot portion for analysis.

(*b*) Do not accept the color which sometimes appears after the addition of the first few drops of bromide-bromate solution as an indication that an excess of bromine has been added. This color may be due to the inhibitor and other non-volatile constituents.

(*c*) If the chloroform residue is so dark in color that the color change due to excess bromine cannot be detected, add an amount of bromide-bromate solution dependent upon the probable dimer content of the sample as indicated in the following table:

Estimated Dimer Content, per cent by weight	Volume of 0.5 *N* Bromide-Bromate Solution, ml.
Less than 0.1	4.0
0.1 to 0.2, incl.	6.0
0.2 to 0.4, incl.	11.0

Calculation

8. Calculate the dimer content of the sample by means of the following equation:

$$\text{Dimer, per cent by weight} = \frac{799.2\,(V_1N_1 - V_2N_2)}{286\,Sd}$$

where:

V_1 = volume of bromide-bromate solution in milliliters,

N_1 = normality of bromide-bromate solution,

V_2 = volume of standard sodium thiosulfate solution in milliliters,

N_2 = normality of standard sodium thiosulfate solution,

S = volume of sample in milliliters, and

d = density of butadiene at the temperature of measurement in grams per milliter.

NOTE 3.—The value 286 is an empirical factor representative of average values obtained on bromination of pure vinyl cyclohexene (theoretical bromine number = 295.5). The operator may desire to determine the titer using pure vinyl-1-cyclohexene in the bromination procedure.

Precision

9. Results should not differ from the mean by more than the following amount:

Repeatability, One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
0.005	...

APPENDIX IV

PROPOSED METHOD OF TEST FOR NONVOLATILE RESIDUE OF POLYMERIZATION GRADE BUTADIENE[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test describes a procedure for the determination of nonvolatile material in polymerization grade butadiene.

Outline of Method

2. A measured volume of liquid butadiene is allowed to evaporate at room temperature from a small glass evaporating dish until only residue remains. The evaporation is then completed by heating the dish to a constant weight at 105 C.

Apparatus

3. (*a*) *Evaporating Dish*, glass, 80 mm. in diameter and 45 mm. in height.

(*b*) *Graduated Cylinder*, capacity of 25 ml., graduated in 0.2-ml. subdivisions, accurate to 0.11 ml.

(*c*) *Oven*, capable of maintaining a temperature of 105 ± 5C.

(*d*) *Cooling Vessel*, tightly covered, such as a glass desiccator, for cooling the evaporating dishes before weighing.

(*e*) *Thermometer*, range of −40 to 50 C., graduated in 1 C. intervals, mercury-filled.

Preparation of Apparatus

4. Clean the evaporating dishes (including a tare) with chromic-acid solution before each test. Remove the dishes from the cleaning solution with stainless-steel forceps and handle only with forceps thereafter. Wash the dishes thoroughly, first with tap water, then with distilled water, and dry in an oven at 105 C. for about 1 hr., or until constant weight is obtained. Before weighing, cool the dishes, for at least 30 min., in the cooling vessel placed near the balance.

Procedure

5. (*a*) Weigh the evaporating dish differentially to the nearest 0.1 mg., using a second dish as a tare on the right-hand pan of the balance.

(*b*) Chill the evaporating dish to ice temperature. Cool the sample and the graduate cylinder to about −20C. Determine the sample temperature to the nearest 1C. and transfer 25 ± 0.2 ml. from the graduate to the evaporating dish. Record volume and temperature.

(*c*) Allow the sample to evaporate at room temperature in a well-ventilated hood. When evaporation is complete, place the evaporating dish (and the tare) in an oven at 105 ± 5 C. until a weight constant to 0.1 mg. is obtained.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1948.

[2] This method was developed and has been cooperatively tested by the Butadiene Producer's Committee on Specifications and Methods of Analysis of the Office of Rubber Reserve. It appears in the Butadiene Laboratory Manual, Office of Rubber Reserve, as Method No. 2.1.56.2.

Before each weighing, cool the dish and the tare for at least 30 min. in the cooling vessel placed near the balance and weigh to the nearest 0.1 mg. against the tare. Between each two weighings, place the dish in the oven at 105 C. for at least 30 min.

Calculation

6. Calculate the amount of nonvolatile residue by means of the following equation:

$$\text{Nonvolatile residue, per cent by weight} = \frac{(B - A)}{Sd} \times 100$$

where:

A = weight of the evaporating dish in grams,

B = weight of the evaporating dish plus residue in grams,

S = volume of the liquid butadiene sample in milliliters, and

d = density of the sample at the temperature of measurement, in grams per milliliter.

Precision

7. Results should not differ from the mean by more than the following amounts:

Repeatability, One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
0.02	0.04

APPENDIX V

PROPOSED FACTORS AND TABLES FOR VOLUME CORRECTION AND SPECIFIC GRAVITY[1] CONVERSION OF LIQUEFIED PETROLEUM GASES[2]

These proposed factors and tables are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

The factors for correcting measured liquid volume to standard volume at 60 F., and the specific gravity at 60 F. corresponding to that obtained at other temperatures for liquefied petroleum gases were determined by the Natural Gasoline Association of America.

An unabridged table for the conversion of an observed specific gravity to the corresponding specific gravity at 60 F. is given in Table I.

The volume correction factors in an unabridged form are given in Table II.

Since the A.S.T.M. Standard D 206 - 36, Abridged Volume Correction Table for Petroleum Oils does not extend beyond 100 deg. A.P.I. (specific gravity at 60 F./60 F. of 0.6112) the accompanying Tables I and II start at this gravity and go to 205.4 deg. A.P.I. (specific gravity at 60 F./60 F. of 0.42).

In Table I the specific gravity at 60 F. is read corresponding to the specific gravity determined at any temperature. For example, having determined a specific gravity of 0.540 at 26 F., the specific gravity at 60 F./60 F. will be found by moving down the column headed 0.540 in Table I until the horizontal line intersects at 26 F. The specific gravity at 60 F./60 F. is read as 0.514.

The liquid volume correction factors in Table II were obtained from the following formula:

$$\text{Volume Correction Factor} = \frac{\text{density at temp. } t}{\text{density at 60 F.}} = \frac{\text{Volume at 60 F.}}{\text{Volume at temp. } t}$$

The values given in Table II are in the form of multipliers; that is, the actual volume measured at an indicated temperature is multiplied by the corresponding factor in the table to secure the volume at 60 F. For example, given a specific gravity at 60 F./60 F. of 0.514 and a liquid temperature of 26 F., the volume correction factor to convert the measured volume at 26 F. to the volume at 60 F. is found by moving down the column headed 0.513 to 0.517 in Table II until it intersects the horizontal line at 26 F. The correction factor read is 1.051. The measured volume is multiplied by this factor to get the volume at 60 F.

---

[1] These tables are derived from density values "in vacuo" and therefore should properly be called "absolute specific gravity."

[2] These proposed factors and tables are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1948.

TABLE I.—CONVERSION OF SPECIFIC GRAVITY TO 60 F.

Observed Temp., deg. F	OBSERVED SPECIFIC GRAVITY															
	0.420	0.425	0.430	0.435	0.440	0.445	0.450	0.455	0.460	0.465	0.470	0.475	0.480	0.485	0.490	0.495
	CORRESPONDING SPECIFIC GRAVITIES AT 60 F./60 F.															
50	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.480	0.485
51	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.481	0.486
52	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.482	0.487
53	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.483	0.488
54	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.484	0.489
55	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.485	0.490
56	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.486	0.491
57	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.487	0.492
58	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.488	0.493
59	...	...	...	...	...	...	...	...	...	...	...	...	...	...	0.489	0.494
60	...	...	...	...	...	...	...	...	...	...	...	...	0.480	0.485	0.490	0.495
61	...	...	...	...	...	...	...	...	...	...	...	...	0.481	0.486	0.491	0.496
62	...	...	...	...	...	...	...	...	...	...	...	...	0.482	0.487	0.492	0.497
63	...	...	...	...	...	...	...	...	...	...	...	...	0.483	0.488	0.493	0.498
64	...	...	...	...	...	...	...	...	...	...	...	...	0.484	0.489	0.494	0.498
65	...	...	...	...	...	...	...	...	...	...	...	...	0.485	0.490	0.494	0.499
66	...	...	...	...	...	...	...	...	...	...	...	...	0.485	0.490	0.495	0.500
67	...	...	...	...	...	...	...	...	...	...	...	...	0.486	0.491	0.496	0.501
68	...	...	...	...	...	...	...	...	...	...	...	...	0.487	0.492	0.497	0.502
69	...	...	...	...	...	...	...	...	...	...	...	...	0.488	0.493	0.498	0.503
70	...	...	...	...	...	...	...	...	...	...	0.479	0.484	0.489	0.494	0.499	0.504
71	...	...	...	...	...	...	...	...	...	...	0.480	0.485	0.490	0.495	0.500	0.505
72	...	...	...	...	...	...	...	...	...	...	0.481	0.486	0.491	0.496	0.501	0.506
73	...	...	...	...	...	...	...	...	...	...	0.482	0.487	0.492	0.497	0.501	0.506
74	...	...	...	...	...	...	...	...	...	...	0.483	0.488	0.493	0.497	0.502	0.507
75	...	...	...	...	...	...	...	...	...	...	0.484	0.488	0.493	0.498	0.503	0.508
76	...	...	...	...	...	...	...	...	...	...	0.484	0.489	0.494	0.499	0.504	0.509
77	...	...	...	...	...	...	...	...	...	...	0.485	0.490	0.495	0.500	0.504	0.509
78	...	...	...	...	...	...	...	...	...	...	0.486	0.491	0.496	0.501	0.505	0.510
79	...	...	...	...	...	...	...	...	...	...	0.487	0.492	0.497	0.502	0.506	0.511
80	...	...	...	...	...	...	...	...	0.479	0.485	0.488	0.493	0.498	0.503	0.507	0.512
81	...	...	...	...	...	...	...	...	0.480	0.486	0.489	0.494	0.499	0.503	0.508	0.513
82	...	...	...	...	...	...	...	...	0.481	0.486	0.490	0.495	0.500	0.504	0.509	0.514
83	...	...	...	...	...	...	...	...	0.482	0.487	0.491	0.496	0.501	0.505	0.510	0.514
84	...	...	...	...	...	...	...	...	0.483	0.488	0.492	0.497	0.501	0.506	0.510	0.515
85	...	...	...	...	...	...	...	...	0.484	0.489	0.493	0.498	0.502	0.507	0.511	0.516
86	...	...	...	...	...	...	...	...	0.484	0.490	0.493	0.498	0.503	0.507	0.512	0.517
87	...	...	...	...	...	...	...	...	0.485	0.491	0.494	0.499	0.504	0.508	0.513	0.517
88	...	...	...	...	...	...	...	...	0.486	0.491	0.495	0.500	0.504	0.509	0.513	0.518
89	...	...	...	...	...	...	...	...	0.487	0.492	0.496	0.501	0.505	0.510	0.514	0.519
90	...	...	...	...	...	...	0.479	0.484	0.488	0.493	0.497	0.502	0.506	0.511	0.515	0.520
91	...	...	...	...	...	...	0.480	0.485	0.489	0.494	0.498	0.503	0.507	0.512	0.516	0.521
92	...	...	...	...	...	...	0.481	0.486	0.490	0.495	0.499	0.503	0.508	0.512	0.517	0.521
93	...	...	...	...	...	...	0.482	0.486	0.491	0.495	0.500	0.504	0.509	0.513	0.518	0.522
94	...	...	...	...	...	...	0.483	0.487	0.492	0.496	0.500	0.505	0.509	0.514	0.518	0.523
95	...	...	...	...	...	...	0.484	0.488	0.492	0.497	0.501	0.506	0.510	0.515	0.519	0.524
96	...	...	...	...	...	...	0.484	0.489	0.493	0.498	0.502	0.507	0.511	0.515	0.520	0.524
97	...	...	...	...	...	...	0.485	0.490	0.494	0.499	0.503	0.507	0.512	0.516	0.521	0.525
98	...	...	...	...	...	...	0.486	0.491	0.495	0.499	0.503	0.508	0.512	0.517	0.521	0.526
99	...	...	...	...	...	...	0.487	0.491	0.496	0.500	0.504	0.509	0.513	0.518	0.522	0.527
100	...	...	...	...	0.480	0.484	0.488	0.492	0.497	0.501	0.505	0.510	0.514	0.519	0.525	0.527
105	...	...	...	...	0.485	0.489	0.493	0.497	0.502	0.505	0.510	0.514	0.518	0.522	0.527	0.531
110	...	...	0.481	0.486	0.489	0.494	0.497	0.502	0.506	0.510	0.514	0.518	0.522	0.526	0.530	0.535
115	...	...	0.485	0.489	0.493	0.497	0.501	0.505	0.510	0.514	0.518	0.522	0.526	0.530	0.534	0.538
120	0.482	0.485	0.489	0.493	0.497	0.501	0.505	0.509	0.513	0.517	0.521	0.525	0.529	0.534	0.538	0.542
125	0.486	0.490	0.494	0.498	0.502	0.505	0.509	0.513	0.517	0.521	0.525	0.529	0.533	0.537	0.541	0.545
130	0.491	0.494	0.498	0.502	0.506	0.510	0.514	0.518	0.521	0.525	0.529	0.533	0.537	0.541	0.545	0.549
135	0.495	0.499	0.502	0.506	0.510	0.514	0.517	0.521	0.525	0.529	0.533	0.537	0.541	0.545	0.549	0.553
140	0.499	0.503	0.506	0.510	0.514	0.517	0.521	0.525	0.529	0.532	0.536	0.540	0.544	0.548	0.552	0.556
	EQUIVALENT A.P.I. GRAVITY															
...	205.4	201.4	197.6	193.8	190.1	186.5	182.9	179.5	176.1	172.8	169.5	166.4	163.3	160.3	157.3	154.4

TABLE I.—CONVERSION OF SPECIFIC GRAVITY TO 60 F. (*continued*).

Observed Temp., deg. F.	Observed Specific Gravity											
	0.500	0.505	0.510	0.515	0.520	0.525	0.530	0.535	0.540	0.545	0.550	0.555
	Corresponding Specific Gravities at 60 F./60 F.											
−50	...	...	...	...	...	...	...	...	...	...	...	...
−45	...	...	...	...	...	...	...	...	...	...	...	...
−40	...	...	...	...	...	...	...	...	...	...	...	...
−35	...	...	...	...	...	...	...	...	...	...	...	...
−30	...	...	...	...	...	...	...	...	...	...	...	0.486
−25	...	...	...	...	...	...	...	...	...	...	...	0.490
−20	...	...	...	...	...	...	...	...	...	0.482	0.488	0.494
−15	...	...	...	...	...	...	...	...	...	0.486	0.492	0.498
−10	...	...	...	...	...	...	...	...	0.484	0.490	0.496	0.502
−5	...	...	...	...	...	...	...	...	0.488	0.494	0.500	0.506
0	...	...	...	...	...	...	0.481	0.487	0.493	0.499	0.505	0.511
1	...	...	...	...	...	...	0.482	0.488	0.494	0.499	0.505	0.511
2	...	...	...	...	...	...	0.483	0.489	0.495	0.500	0.506	0.512
3	...	...	...	...	...	...	0.484	0.490	0.495	0.501	0.507	0.513
4	...	...	...	...	...	...	0.484	0.490	0.496	0.502	0.508	0.513
5	...	...	...	...	...	...	0.485	0.491	0.497	0.503	0.508	0.514
6	...	...	...	...	...	...	0.486	0.492	0.498	0.503	0.509	0.515
7	...	...	...	...	...	...	0.487	0.493	0.498	0.504	0.510	0.516
8	...	...	...	...	...	...	0.487	0.493	0.499	0.505	0.511	0.516
9	...	...	...	...	...	...	0.488	0.494	0.500	0.506	0.511	0.517
10	...	...	...	...	...	0.484	0.489	0.495	0.501	0.506	0.512	0.518
11	...	...	...	...	...	0.485	0.490	0.496	0.502	0.507	0.513	0.519
12	...	...	...	...	...	0.486	0.491	0.497	0.503	0.508	0.514	0.519
13	...	...	...	...	...	0.486	0.491	0.497	0.503	0.509	0.514	0.520
14	...	...	...	...	...	0.487	0.492	0.498	0.504	0.510	0.515	0.521
15	...	...	...	...	...	0.488	0.493	0.499	0.505	0.510	0.516	0.522
16	...	...	...	...	...	0.489	0.494	0.500	0.506	0.511	0.517	0.522
17	...	...	...	...	...	0.490	0.495	0.501	0.506	0.512	0.518	0.523
18	...	...	...	...	...	0.491	0.495	0.501	0.507	0.513	0.518	0.524
19	...	...	...	...	...	0.491	0.496	0.502	0.508	0.514	0.519	0.525
20	...	...	...	0.481	0.486	0.492	0.497	0.503	0.509	0.514	0.520	0.526
21	...	...	...	0.482	0.487	0.493	0.498	0.504	0.510	0.515	0.521	0.526
22	...	...	...	0.483	0.488	0.494	0.499	0.505	0.511	0.516	0.522	0.527
23	...	...	...	0.484	0.489	0.495	0.500	0.506	0.511	0.517	0.522	0.528
24	...	...	...	0.485	0.490	0.496	0.501	0.507	0.512	0.518	0.523	0.529
25	...	...	...	0.486	0.491	0.496	0.502	0.508	0.513	0.519	0.524	0.530
26	...	...	...	0.486	0.491	0.497	0.502	0.508	0.514	0.519	0.525	0.530
27	...	...	...	0.487	0.492	0.498	0.503	0.509	0.515	0.520	0.526	0.531
28	...	...	...	0.488	0.493	0.499	0.504	0.510	0.515	0.521	0.526	0.532
29	...	...	...	0.489	0.494	0.500	0.505	0.511	0.516	0.522	0.527	0.533
30	...	...	0.484	0.490	0.495	0.501	0.506	0.512	0.517	0.523	0.528	0.534
31	...	...	0.485	0.491	0.496	0.501	0.507	0.513	0.518	0.523	0.529	0.534
32	...	...	0.486	0.492	0.497	0.502	0.508	0.513	0.519	0.524	0.530	0.535
33	...	...	0.487	0.492	0.498	0.503	0.509	0.514	0.520	0.525	0.531	0.536
34	...	...	0.488	0.493	0.499	0.504	0.509	0.515	0.520	0.526	0.531	0.536
35	...	...	0.488	0.494	0.499	0.505	0.510	0.516	0.521	0.526	0.532	0.537
36	...	...	0.489	0.495	0.500	0.506	0.511	0.517	0.522	0.527	0.533	0.538
37	...	...	0.490	0.496	0.501	0.507	0.512	0.517	0.523	0.528	0.533	0.539
38	...	...	0.491	0.497	0.502	0.507	0.512	0.518	0.523	0.529	0.534	0.539
39	...	...	0.492	0.498	0.503	0.508	0.513	0.519	0.524	0.529	0.535	0.540
40	0.483	0.488	0.493	0.499	0.504	0.509	0.514	0.520	0.525	0.530	0.536	0.541
41	0.484	0.489	0.494	0.500	0.505	0.510	0.515	0.521	0.526	0.531	0.537	0.542
42	0.485	0.490	0.495	0.500	0.506	0.511	0.516	0.521	0.527	0.532	0.537	0.542
43	0.485	0.491	0.496	0.501	0.506	0.512	0.517	0.522	0.527	0.533	0.538	0.543
44	0.486	0.492	0.497	0.502	0.507	0.512	0.517	0.523	0.528	0.533	0.539	0.544
45	0.487	0.492	0.497	0.503	0.508	0.513	0.518	0.524	0.529	0.534	0.539	0.544
46	0.488	0.493	0.498	0.504	0.509	0.514	0.519	0.524	0.530	0.535	0.540	0.545
47	0.489	0.494	0.499	0.504	0.510	0.515	0.520	0.525	0.530	0.536	0.541	0.546
48	0.489	0.495	0.500	0.505	0.510	0.515	0.520	0.526	0.531	0.536	0.542	0.547
49	0.490	0.496	0.501	0.506	0.511	0.516	0.521	0.527	0.532	0.537	0.542	0.547

TABLE I.—CONVERSION OF SPECIFIC GRAVITY TO 60 F. (*Continued*).

OBSERVED SPECIFIC GRAVITY											Observed Temp., deg. F.
0.560	0.565	0.570	0.575	0.580	0.585	0.590	0.595	0.600	0.605	0.610	
CORRESPONDING SPECIFIC GRAVITIES AT 60 F./60 F.											
...	0.481	0.487	0.494	0.500	0.507	0.513	0.519	0.526	0.532	0.538	−50
...	0.485	0.491	0.497	0.504	0.511	0.517	0.523	0.529	0.536	0.542	−45
0.483	0.489	0.495	0.502	0.508	0.514	0.521	0.527	0.533	0.539	0.545	−40
0.487	0.494	0.500	0.506	0.512	0.518	0.525	0.531	0.537	0.543	0.549	−35
0.491	0.498	0.504	0.510	0.516	0.523	0.529	0.535	0.541	0.547	0.552	−30
0.496	0.502	0.508	0.514	0.520	0.526	0.532	0.538	0.544	0.550	0.556	−25
0.500	0.506	0.512	0.518	0.524	0.530	0.536	0.542	0.548	0.553	0.559	−20
0.504	0.510	0.516	0.522	0.528	0.534	0.540	0.546	0.552	0.557	0.563	−15
0.508	0.514	0.520	0.526	0.532	0.537	0.543	0.549	0.555	0.561	0.566	−10
0.512	0.518	0.524	0.530	0.536	0.041	0.547	0.553	0.559	0.564	0.570	−5
0.516	0.522	0.528	0.534	0.540	0.545	0.551	0.556	0.562	0.568	0.573	0
0.517	0.523	0.529	0.534	0.540	0.546	0.551	0.557	0.563	0.568	0.574	1
0.517	0.524	0.529	0.535	0.541	0.546	0.552	0.558	0.563	0.569	0.574	2
0.518	0.524	0.530	0.536	0.542	0.547	0.553	0.558	0.564	0.569	0.575	3
0.519	0.525	0.531	0.536	0.542	0.548	0.553	0.559	0.564	0.570	0.576	4
0.520	0.526	0.531	0.537	0.543	0.548	0.554	0.560	0.565	0.571	0.576	5
0.520	0.526	0.532	0.538	0.544	0.549	0.555	0.560	0.566	0.571	0.577	6
0.521	0.527	0.533	0.538	0.544	0.550	0.555	0.561	0.567	0.572	0.578	7
0.522	0.528	0.534	0.539	0.545	0.550	0.556	0.562	0.567	0.573	0.579	8
0.523	0.528	0.534	0.540	0.545	0.551	0.557	0.562	0.568	0.573	0.579	9
0.523	0.529	0.535	0.540	0.546	0.552	0.557	0.563	0.568	0.574	0.580	10
0.524	0.530	0.535	0.541	0.547	0.552	0.558	0.564	0.569	0.575	0.580	11
0.525	0.531	0.536	0.542	0.547	0.553	0.558	0.564	0.569	0.575	0.581	12
0.526	0.531	0.537	0.542	0.548	0.554	0.559	0.565	0.570	0.576	0.582	13
0.526	0.532	0.538	0.543	0.549	0.555	0.560	0.566	0.571	0.577	0.582	14
0.527	0.533	0.538	0.544	0.550	0.555	0.561	0.566	0.572	0.577	0.583	15
0.528	0.534	0.539	0.545	0.550	0.556	0.561	0.567	0.572	0.578	0.584	16
0.529	0.534	0.540	0.545	0.551	0.557	0.562	0.568	0.573	0.579	0.584	17
0.529	0.535	0.541	0.546	0.552	0.557	0.563	0.568	0.574	0.579	0.585	18
0.530	0.536	0.541	0.547	0.553	0.558	0.564	0.569	0.575	0.580	0.586	19
0.531	0.537	0.542	0.548	0.553	0.559	0.564	0.570	0.575	0.581	0.586	20
0.532	0.538	0.543	0.548	0.554	0.559	0.565	0.570	0.576	0.581	0.587	21
0.533	0.538	0.544	0.549	0.554	0.560	0.565	0.571	0.576	0.582	0.587	22
0.533	0.539	0.544	0.550	0.555	0.561	0.566	0.572	0.577	0.583	0.588	23
0.534	0.540	0.545	0.551	0.556	0.561	0.567	0.572	0.578	0.583	0.588	24
0.535	0.541	0.546	0.551	0.557	0.562	0.568	0.573	0.578	0.584	0.589	25
0.536	0.541	0.547	0.552	0.557	0.563	0.568	0.574	0.579	0.584	0.590	26
0.537	0.542	0.547	0.553	0.558	0.564	0.569	0.574	0.580	0.585	0.590	27
0.537	0.543	0.548	0.554	0.559	0.564	0.570	0.575	0.581	0.586	0.591	28
0.538	0.544	0.549	0.554	0.560	0.565	0.570	0.576	0.581	0.586	0.591	29
0.539	0.544	0.550	0.555	0.560	0.566	0.571	0.576	0.582	0.587	0.592	30
0.540	0.545	0.550	0.556	0.561	0.566	0.572	0.577	0.582	0.587	0.593	31
0.540	0.546	0.551	0.556	0.561	0.567	0.572	0.578	0.583	0.588	0.593	32
0.541	0.547	0.552	0.557	0.562	0.568	0.573	0.578	0.584	0.589	0.594	33
0.542	0.547	0.553	0.558	0.563	0.568	0.574	0.579	0.584	0.589	0.595	34
0.543	0.548	0.553	0.559	0.564	0.569	0.574	0.580	0.585	0.590	0.595	35
0.543	0.549	0.554	0.559	0.564	0.570	0.575	0.580	0.586	0.591	0.596	36
0.544	0.549	0.555	0.560	0.565	0.570	0.576	0.581	0.586	0.591	0.597	37
0.545	0.550	0.556	0.561	0.566	0.571	0.577	0.582	0.587	0.592	0.598	38
0.545	0.551	0.556	0.561	0.567	0.572	0.577	0.582	0.587	0.593	0.598	39
0.546	0.551	0.557	0.562	0.567	0.572	0.578	0.583	0.588	0.593	0.599	40
0.547	0.552	0.557	0.563	0.568	0.573	0.578	0.584	0.589	0.594	0.599	41
0.547	0.553	0.558	0.563	0.568	0.574	0.579	0.584	0.589	0.595	0.600	42
0.548	0.554	0.559	0.564	0.569	0.574	0.580	0.585	0.590	0.595	0.600	43
0.549	0.554	0.559	0.565	0.570	0.575	0.580	0.586	0.591	0.596	0.601	44
0.550	0.555	0.560	0.565	0.571	0.576	0.581	0.586	0.591	0.597	0.602	45
0.550	0.556	0.561	0.566	0.571	0.577	0.582	0.587	0.592	0.597	0.603	46
0.551	0.556	0.561	0.567	0.572	0.577	0.582	0.588	0.593	0.598	0.603	47
0.552	0.557	0.562	0.567	0.573	0.578	0.583	0.588	0.594	0.599	0.604	48
0.553	0.558	0.563	0.568	0.573	0.579	0.584	0.589	0.594	0.599	0.604	49

TABLE I.—CONVERSION OF SPECIFIC GRAVITY TO 60 F. (*Continued*).

Observed Temp., deg. F.	OBSERVED SPECIFIC GRAVITY											
	0.500	0.505	0.510	0.515	0.520	0.525	0.530	0.535	0.540	0.545	0.550	0.555
	CORRESPONDING SPECIFIC GRAVITIES AT 60 F./60 F.											
50	0.491	0.497	0.502	0.507	0.512	0.517	0.522	0.527	0.533	0.538	0.543	0.548
51	0.492	0.497	0.503	0.508	0.513	0.518	0.523	0.528	0.533	0.538	0.544	0.549
52	0.493	0.498	0.504	0.509	0.514	0.519	0.524	0.529	0.534	0.539	0.544	0.549
53	0.494	0.499	0.504	0.509	0.515	0.520	0.525	0.530	0.535	0.540	0.545	0.550
54	0.494	0.500	0.505	0.510	0.515	0.520	0.525	0.530	0.536	0.541	0.546	0.551
55	0.495	0.501	0.506	0.511	0.516	0.521	0.526	0.531	0.536	0.541	0.546	0.552
56	0.496	0.502	0.507	0.512	0.517	0.522	0.527	0.532	0.537	0.542	0.547	0.552
57	0.497	0.503	0.507	0.513	0.518	0.523	0.528	0.533	0.538	0.543	0.548	0.553
58	0.498	0.503	0.508	0.513	0.518	0.523	0.529	0.534	0.539	0.544	0.549	0.554
59	0.499	0.504	0.509	0.514	0.519	0.524	0.529	0.534	0.539	0.544	0.549	0.554
60	0.500	0.505	0.510	0.515	0.520	0.525	0.530	0.535	0.540	0.545	0.550	0.555
61	0.501	0.506	0.511	0.516	0.521	0.526	0.531	0.536	0.541	0.546	0.551	0.556
62	0.502	0.507	0.512	0.517	0.522	0.527	0.532	0.537	0.541	0.546	0.551	0.556
63	0.503	0.508	0.513	0.517	0.522	0.527	0.532	0.537	0.542	0.547	0.552	0.557
64	0.504	0.508	0.513	0.518	0.523	0.528	0.533	0.538	0.543	0.548	0.553	0.558
65	0.504	0.509	0.514	0.519	0.524	0.529	0.534	0.539	0.544	0.549	0.554	0.559
66	0.505	0.510	0.515	0.520	0.525	0.530	0.535	0.540	0.544	0.549	0.554	0.559
67	0.506	0.511	0.516	0.521	0.526	0.530	0.535	0.540	0.545	0.550	0.555	0.560
68	0.507	0.512	0.517	0.521	0.526	0.531	0.536	0.541	0.546	0.551	0.556	0.561
69	0.508	0.513	0.518	0.522	0.527	0.532	0.537	0.542	0.547	0.552	0.557	0.561
70	0.509	0.513	0.518	0.523	0.428	0.533	0.538	0.543	0.547	0.552	0.557	0.562
71	0.510	0.514	0.519	0.524	0.529	0.533	0.538	0.543	0.548	0.553	0.558	0.563
72	0.511	0.515	0.520	0.525	0.529	0.534	0.539	0.544	0.548	0.553	0.558	0.563
73	0.511	0.516	0.521	0.525	0.530	0.535	0.540	0.545	0.549	0.554	0.559	0.564
74	0.512	0.517	0.521	0.526	0.531	0.536	0.541	0.545	0.550	0.555	0.560	0.565
75	0.513	0.517	0.522	0.527	0.532	0.536	0.541	0.546	0.551	0.556	0.561	0.565
76	0.513	0.518	0.523	0.527	0.532	0.537	0.542	0.547	0.551	0.556	0.561	0.566
77	0.514	0.519	0.524	0.528	0.533	0.538	0.543	0.547	0.552	0.557	0.562	0.567
78	0.515	0.520	0.524	0.529	0.534	0.539	0.544	0.548	0.553	0.558	0.563	0.567
79	0.516	0.520	0.525	0.530	0.535	0.540	0.544	0.549	0.554	0.558	0.563	0.568
80	0.517	0.521	0.526	0.530	0.535	0.540	0.545	0.549	0.554	0.559	0.564	0.569
81	0.518	0.522	0.527	0.531	0.536	0.541	0.545	0.550	0.555	0.560	0.565	0.569
82	0.519	0.523	0.528	0.532	0.537	0.541	0.546	0.551	0.555	0.560	0.565	0.570
83	0.519	0.524	0.528	0.533	0.538	0.542	0.547	0.552	0.556	0.561	0.566	0.571
84	0.520	0.524	0.529	0.534	0.538	0.543	0.548	0.552	0.557	0.562	0.567	0.571
85	0.521	0.525	0.530	0.534	0.539	0.544	0.548	0.553	0.558	0.563	0.568	0.572
86	0.521	0.526	0.531	0.535	0.540	0.544	0.549	0.554	0.558	0.563	0.568	0.573
87	0.522	0.527	0.531	0.536	0.541	0.545	0.550	0.554	0.559	0.564	0.569	0.573
88	0.523	0.527	0.532	0.537	0.541	0.546	0.551	0.555	0.560	0.564	0.569	0.574
89	0.524	0.528	0.533	0.537	0.542	0.547	0.551	0.556	0.561	0.565	0.570	0.575
90	0.524	0.529	0.534	0.538	0.543	0.547	0.552	0.557	0.561	0.566	0.570	0.575
91	0.525	0.530	0.534	0.539	0.543	0.548	0.553	0.557	0.562	0.566	0.571	0.576
92	0.526	0.530	0.535	0.540	0.544	0.549	0.553	0.558	0.562	0.567	0.572	0.576
93	0.527	0.531	0.536	9.540	0.545	0.549	0.554	0.559	0.563	0.568	0.572	0.577
94	0.528	0.532	0.537	0.541	0.545	0.550	0.554	0.559	0.563	0.568	0.573	0.578
95	0.528	0.533	0.537	0.542	0.546	0.551	0.555	0.560	0.564	0.569	0.574	0.578
96	0.529	0.533	0.538	0.542	0.547	0.551	0.556	0.561	0.565	0.569	0.574	0.579
97	0.530	0.534	0.539	0.543	0.547	0.552	0.556	0.561	0.566	0.570	0.575	0.580
98	0.531	0.535	0.540	0.543	0.548	0.552	0.557	0.562	0.566	0.571	0.575	0.580
99	0.531	0.535	0.540	0.544	0.548	0.553	0.558	0.563	0.567	0.571	0.576	0.581
100	0.532	0.536	0.541	0.545	0.549	0.554	0.558	0.563	0.567	0.572	0.577	0.581
105	0.535	0.540	0.544	0.548	0.553	0.557	0.562	0.566	0.571	0.575	0.580	0.584
110	0.539	0.543	0.548	0.552	0.557	0.561	0.565	0.570	0.574	0.579	0.583	0.587
115	0.543	0.547	0.551	0.555	0.560	0.564	0.568	0.573	0.577	0.582	0.586	0.590
120	0.546	0.550	0.554	0.559	0.563	0.567	0.571	0.576	0.580	0.585	0.589	0.594
125	0.550	0.554	0.558	0.562	0.566	0.570	0.574	0.579	0.583	0.588	0.592	0.596
130	0.553	0.557	0.561	0.565	0.569	0.573	0.577	0.582	0.586	0.590	0.595	0.599
135	0.557	0.561	0.565	0.569	0.573	0.577	0.581	0.585	0.589	0.593	0.598	0.602
140	0.560	0.564	0.568	0.572	0.576	0.580	0.584	0.588	0.592	0.596	0.601	0.605
	EQUIVALENT A.P.I. GRAVITY											
...	151.5	148.6	145.9	143.2	140.6	138.0	135.5	133.0	130.5	128.1	125.8	123.4

TABLE I.—CONVERSION OF SPECIFIC GRAVITY TO 60 F. (*Concluded*).

OBSERVED SPECIFIC GRAVITY											Observed Temp., deg. F.
0.560	0.565	0.570	0.575	0.580	0.585	0.590	0.595	0.600	0.605	0.610	
CORRESPONDING SPECIFIC GRAVITIES AT 60 F./60 F.											
0.553	0.558	0.563	0.569	0.574	0.579	0.584	0.589	0.595	0.600	0.605	50
0.554	0.559	0.564	0.569	0.575	0.580	0.585	0.590	0.595	0.600	0.605	51
0.554	0.560	0.564	0.570	0.575	0.580	0.585	0.591	0.596	0.601	0.606	52
0.555	0.560	0.565	0.571	0.576	0.581	0.586	0.591	0.596	0.601	0.606	53
0.556	0.561	0.566	0.571	0.576	0.581	0.586	0.592	0.597	0.602	0.607	54
0.557	0.562	0.567	0.572	0.577	0.582	0.587	0.592	0.597	0.602	0.607	55
0.557	0.562	0.567	0.573	0.578	0.583	0.588	0.593	0.598	0.603	0.608	56
0.558	0.563	0.568	0.573	0.578	0.583	0.588	0.593	0.598	0.603	0.608	57
0.559	0.564	0.569	0.574	0.579	0.584	0.589	0.594	0.599	0.604	0.609	58
0.559	0.564	0.569	0.574	0.579	0.584	0.589	0.594	0.600	0.605	0.610	59
0.560	0.565	0.570	0.575	0.580	0.585	0.590	0.595	0.600	0.605	0.610	60
0.561	0.566	0.571	0.576	0.581	0.586	0.591	0.596	0.601	0.606	0.611	61
0.561	0.566	0.571	0.576	0.581	0.586	0.591	0.596	0.601	0.606	0.611	62
0.562	0.567	0.572	0.577	0.582	0.587	0.592	0.597	0.602	0.607	0.612	63
0.563	0.568	0.573	0.578	0.582	0.587	0.592	0.597	0.602	0.607	0.612	64
0.564	0.568	0.573	0.578	0.583	0.588	0.593	0.598	0.603	0.608	0.613	65
0.564	0.569	0.574	0.579	0.584	0.589	0.594	0.599	0.604	0.609	0.614	66
0.565	0.570	0.575	0.580	0.584	0.589	0.594	0.599	0.604	0.609	0.614	67
0.566	0.571	0.576	0.580	0.585	0.590	0.595	0.600	0.605	0.610	0.615	68
0.566	0.571	0.576	0.581	0.586	0.591	0.596	0.600	0.605	0.610	0.615	69
0.567	0.572	0.577	0.582	0.586	0.591	0.596	0.601	0.606	0.611	0.616	70
0.568	0.572	0.577	0.582	0.587	0.592	0.597	0.602	0.607	0.611	0.616	71
0.568	0.573	0.578	0.583	0.587	0.592	0.597	0.602	0.607	0.612	0.617	72
0.569	0.574	0.579	0.583	0.588	0.593	0.598	0.603	0.608	0.613	0.618	73
0.569	0.574	0.579	0.584	0.589	0.594	0.598	0.603	0.608	0.613	0.618	74
0.570	0.575	0.580	0.585	0.590	0.594	0.599	0.604	0.609	0.614	0.619	75
0.571	0.576	0.581	0.585	0.590	0.595	0.600	0.605	0.510	0.615	0.620	76
0.572	0.576	0.581	0.586	0.591	0.596	0.600	0.605	0.610	0.615	0.620	77
0.572	0.577	0.582	0.587	0.592	0.596	0.601	0.606	0.611	0.616	0.621	78
0.573	0.578	0.582	0.587	0.592	0.597	0.602	0.606	0.611	0.616	0.621	79
0.573	0.578	0.583	0.588	0.593	0.597	0.602	0.607	0.612	0.617	0.622	80
0.574	0.579	0.584	0.588	0.593	0.598	0.603	0.608	0.612	0.617	0.622	81
0.574	0.579	0.584	0.589	0.594	0.599	0.603	0.608	0.613	0.618	0.623	82
0.575	0.580	0.585	0.590	0.594	0.599	0.604	0.609	0.614	0.618	0.623	83
0.576	0.581	0.585	0.590	0.595	0.600	0.604	0.609	0.614	0.619	0.624	84
0.577	0.581	0.586	0.591	0.596	0.600	0.605	0.610	0.615	0.620	0.624	85
0.577	0.582	0.587	0.592	0.597	0.601	0.606	0.611	0.616	0.620	0.625	86
0.578	0.583	0.587	0.592	0.597	0.602	0.606	0.611	0.616	0.621	0.626	87
0.579	0.583	0.588	0.593	0.598	0.602	0.607	0.612	0.617	0.621	0.626	88
0.579	0.584	0.589	0.593	0.598	0.603	0.608	0.612	0.617	0.622	0.627	89
0.580	0.585	0.589	0.594	0.599	0.603	0.608	0.613	0.618	0.622	0.627	90
0.580	0.585	0.590	0.595	0.599	0.604	0.609	0.613	0.618	0.623	0.628	91
0.581	0.586	0.590	0.595	0.600	0.605	0.609	0.614	0.619	0.623	0.628	92
0.582	0.586	0.591	0.596	0.600	0.605	0.610	0.615	0.619	0.624	0.629	93
0.582	0.587	0.591	0.596	0.601	0.606	0.610	0.615	0.620	0.624	0.629	94
0.583	0.587	0.592	0.597	0.602	0.606	0.611	0.616	0.620	0.625	0.630	95
0.584	0.588	0.593	0.597	0.602	0.607	0.612	0.616	0.621	0.626	0.630	96
0.584	0.589	0.593	0.598	0.603	0.607	0.612	0.617	0.621	0.626	0.631	97
0.585	0.589	0.594	0.599	0.603	0.608	0.613	0.617	0.622	0.627	0.631	98
0.585	0.590	0.594	0.599	0.604	0.608	0.613	0.618	0.622	0.627	0.632	99
0.586	0.590	0.595	0.600	0.604	0.609	0.614	0.618	0.623	0.628	0.632	100
0.589	0.593	0.598	0.603	...	...	...	...	...	...	...	105
0.592	0.596	...	...	...	...	...	...	...	...	...	110
0.595	0.599	...	...	...	...	...	...	...	...	...	115
0.598	0.603	...	...	...	...	...	...	...	...	...	120
...	...	...	...	...	...	...	...	...	...	...	125
...	...	...	...	...	...	...	...	...	...	...	130
...	...	...	...	...	...	...	...	...	...	...	135
...	...	...	...	...	...	...	...	...	...	...	140
EQUIVALENT A.P.I. GRAVITY											
121.2	118.9	116.7	114.6	112.5	110.4	108.3	106.3	104.3	102.4	100.5	...

TABLE II.—VOLUME CORRECTION FACTORS.

Observed Temp., deg. F.	Specific Gravity at 60 F./60 F.												
	0.498 to 0.502	0.503 to 0.507	Propane to 0.5079	0.508 to 0.512	0.513 to 0.517	0.518 to 0.522	0.523 to 0.527	0.528 to 0.532	0.533 to 0.537	0.538 to 0.542	0.543 to 0.547	0.548 to 0.552	0.553 to 0.557
	Volume Correction Factors												
−50	1.160	1.156	1.155	1.153	1.150	1.146	1.143	1.140	1.137	1.133	1.130	1.127	1.124
−45	1.153	1.150	1.148	1.146	1.144	1.140	1.137	1.134	1.131	1.128	1.125	1.122	1.119
−40	1.147	1.144	1.142	1.140	1.137	1.134	1.131	1.128	1.125	1.122	1.119	1.117	1.114
−35	1.140	1.137	1.135	1.134	1.131	1.128	1.125	1.122	1.119	1.116	1.114	1.112	1.110
−30	1.134	1.131	1.129	1.128	1.125	1.122	1.119	1.116	1.113	1.111	1.108	1.106	1.104
−25	1.127	1.124	1.122	1.121	1.118	1.115	1.113	1.110	1.107	1.105	1.102	1.100	1.098
−20	1.120	1.117	1.115	1.114	1.112	1.109	1.107	1.104	1.102	1.099	1.097	1.095	1.092
−15	1.112	1.110	1.108	1.107	1.105	1.102	1.100	1.097	1.095	1.093	1.091	1.089	1.087
−10	1.105	1.103	1.102	1.100	1.098	1.095	1.093	1.091	1.089	1.087	1.085	1.083	1.081
−5	1.098	1.096	1.094	1.094	1.092	1.089	1.087	1.085	1.083	1.081	1.080	1.078	1.076
0	1.092	1.089	1.088	1.088	1.086	1.084	1.082	1.080	1.078	1.076	1.074	1.073	1.071
1	1.090	1.088	1.087	1.086	1.084	1.083	1.080	1.079	1.077	1.075	1.073	1.072	1.070
2	1.089	1.087	1.086	1.085	1.083	1.081	1.079	1.077	1.076	1.074	1.072	1.070	1.069
3	1.088	1.086	1.084	1.084	1.082	1.080	1.078	1.076	1.075	1.073	1.071	1.069	1.068
4	1.086	1.084	1.083	1.082	1.081	1.079	1.077	1.075	1.073	1.071	1.070	1.068	1.067
5	1.085	1.083	1.082	1.081	1.079	1.077	1.075	1.074	1.072	1.070	1.069	1.067	1.065
6	1.084	1.082	1.080	1.080	1.078	1.076	1.074	1.072	1.071	1.069	1.067	1.065	1.064
7	1.082	1.080	1.079	1.078	1.077	1.075	1.073	1.071	1.070	1.068	1.066	1.064	1.063
8	1.081	1.079	1.078	1.077	1.075	1.074	1.072	1.070	1.068	1.066	1.065	1.063	1.062
9	1.079	1.078	1.077	1 076	1.074	1.072	1.070	1.069	1.067	1.065	1.064	1.062	1.061
10	1.078	1.076	1.075	1.074	1.073	1.071	1.069	1.067	1.066	1.064	1.063	1.061	1.059
11	1.077	1.075	1.074	1.073	1.071	1.070	1.068	1.066	1.064	1.063	1.061	1.060	1.058
12	1.075	1.073	1.072	1.071	1.070	1.068	1.066	1.064	1.063	1.061	1.060	1.059	1.057
13	1.074	1.072	1.071	0.070	1.069	1.067	1.065	1.063	1.062	1 060	1.059	1.057	1.056
14	1.072	1.071	1.070	1.069	1.067	1.066	1.064	1.062	1.061	1.059	1.058	1.056	1.055
15	1.071	1.070	1.069	1.068	1.066	1.064	1.063	1.061	1.059	1.058	1.056	1.055	1.053
16	1.070	1.068	1.067	1.066	1.065	1.063	1.061	1.060	1.058	1.056	1.055	1.054	1.052
17	1.069	1.067	1.066	1.065	1.063	1.062	1.060	1.058	1.057	1.055	1.054	1.052	1.051
18	1.067	1.066	1.065	1.064	1.062	1.061	1.059	1.057	1.056	1.054	1.053	1.051	1.050
19	1.066	1.064	1.063	1.062	1.061	1.059	1.057	1.056	1.054	1.053	1.051	1.050	1.049
20	1.064	1.063	1.062	1.061	1.059	1.058	1.056	1.054	1.053	1.051	1.050	1.049	1.047
21	1.063	1.061	1.060	1.060	1.058	1.056	1.055	1.053	1.052	1.050	1.049	1.048	1.046
22	1.061	1.060	1.059	1.058	1.057	1.055	1.053	1.052	1.051	1.049	1.048	1.046	1.045
23	1.060	1.058	1.057	1.057	1.055	1.053	1.052	1.051	1.049	1.048	1.047	1.045	1.044
24	1.058	1.057	1.056	1.055	1.054	1.052	1.051	1.049	1.048	1.046	1.045	1.044	1.043
25	1.057	1.055	1.054	1.054	1.052	1.050	1.049	1.048	1.047	1.045	1.044	1.043	1.042
26	1.055	1.054	1.053	1.052	1.051	1.049	1.048	1.047	1.045	1.044	1.043	1.042	1.041
27	1.054	1.052	1.051	1.051	1.049	1.048	1.047	1.045	1.044	1.043	1.042	1.041	1.039
28	1.052	1.051	1.050	1.049	1.048	1.047	1.045	1.944	1.043	1.041	1.040	1.039	1.038
29	1.051	1.049	1.048	1.048	1.046	1.045	1.044	1.043	1.042	1.040	1.039	1.038	1.037
30	1.049	1.048	1.047	1.046	1.045	1.044	1.043	1.041	1.040	1.039	1.038	1.037	1.036
31	1.047	1.046	1.045	1.045	1.044	1.042	1.041	1.040	1.039	1.038	1.037	1.036	1.035
32	1.046	1.045	1.044	1.043	1.042	1.041	1.040	1.038	1.037	1.036	1.036	1.035	1.034
33	1.044	1.043	1.042	1.042	1.041	1.040	1.038	1.037	1.036	1.035	1.035	1.034	1.033
34	1.043	1.041	1.041	1.040	1.039	1.038	1.037	1.036	1.035	1.034	1.033	1.032	1.031
35	1.041	1.040	1.039	1.039	1.038	1.037	1.036	1.035	1.034	1.033	1.032	1.031	1.030
36	1.039	1.038	1.038	1.037	1.036	1.035	1.034	1.033	1.032	1.031	1.031	1.030	1.029
37	1.038	1.037	1.036	1.036	1.035	1.033	1.033	1.032	1.031	1.030	1.030	1.029	1.028
38	1.036	1.035	1.035	1.034	1.033	1.032	1.032	1.031	1.030	1.029	1.028	1.027	1.027
39	1.035	1.034	1.033	1.033	1.032	1.031	1.030	1.029	1.029	1.028	1.027	1.026	1.026
40	1.033	1.032	1.032	1.031	1.030	1.029	1.029	1.028	1.027	1.026	1.026	1.025	1.024
41	1.031	1.031	1.030	1.030	1.029	1.028	1.028	1.027	1.026	1.025	1.024	1.024	1.023
42	1.030	1.029	1.029	1.028	1.027	1.027	1.026	1.025	1.025	1.024	1.023	1.023	1.022
43	1.028	1.027	1.027	1.027	1.026	1.025	1.025	1.024	1.023	1.022	1.022	1.021	1.021
44	1.027	1.026	1.026	1.025	1.024	1.023	1.023	1.022	1.022	1.021	1.021	1.020	1.020
45	1.025	1.024	1.024	1.024	1.023	1.022	1.022	1.021	1.020	1.020	1.019	1.019	1.018
46	1.023	1.023	1.022	1.022	1.022	1.021	1.020	1.020	1.019	1.018	1.018	1.018	1.017
47	1.022	1.021	1.021	1.021	1.020	1.019	1.019	1.018	1.018	1.017	1.017	1.016	1.016
48	1.020	1.020	1.019	1.019	1.019	1.018	1.017	1.017	1.016	1.016	1.015	1.015	1.015
49	1.019	1.018	1.018	1.018	1.017	1.017	1.016	1.015	1.015	1.015	1.014	1.014	1.013

TABLE II.—VOLUME CORRECTION FACTORS (*Continued*).

Specific Gravity at 60 F./60 F.													Observed Temp., deg. F.
0.558 to 0.562	Iso-Butane to 0.563	0.563 to 0.567	0.568 to 0.572	0.573 to 0.577	0.578 to 0.582	*n*-Butene to 0.5844	0.583 to 0.587	0.588 to 0.592	0.593 to 0.597	0.598 to 0.602	0.603 to 0.607	0.608 to 0.612	
Volume Correction Factors													
1.122	1.120	1.119	1.116	1.114	1.111	1.108	1.109	1.106	1.104	1.102	1.100	1.098	−50
1.117	1.115	1.114	1.111	1.109	1.106	1.103	1.103	1.101	1.099	1.097	1.095	1.094	−45
1.111	1.110	1.109	1.106	1.104	1.101	1.099	1.099	1.097	1.095	1.093	1.091	1.089	−40
1.106	1.105	1.104	1.101	1.099	1.096	1.094	1.094	1.092	1.090	1.088	1.087	1.085	−35
1.101	1.100	1.098	1.096	1.094	1.092	1.090	1.090	1.088	1.086	1.084	1.083	1.081	−30
1.095	1.094	1.093	1.091	1.089	1.087	1.085	1.085	1.083	1.082	1.080	1.078	1.077	−25
1.090	1.089	1.088	1.086	1.084	1.082	1.080	1.080	1.079	1.077	1.076	1.074	1.073	−20
1.084	1.083	1.082	1.080	1.079	1.077	1.075	1.075	1.074	1.073	1.071	1.070	1.068	−15
1.079	1.078	1.077	1.075	1.074	1.072	1.071	1.071	1.069	1.068	1.066	1.065	1.064	−10
1.074	1.073	1.073	1.070	1.069	1.067	1.066	1.066	1.065	1.063	1.062	1.061	1.059	−5
1.069	1.068	1.068	1.066	1.064	1.063	1.062	1.062	1.061	1.059	1.058	1.057	1.055	0
1.068	1.067	1.067	1.065	1.063	1.062	1.061	1.061	1.060	1.058	1.057	1.056	1.054	1
1.067	1.066	1.066	1.064	1.062	1.061	1.060	1.060	1.059	1.057	1.056	1.055	1.053	2
1.066	1.065	1.064	1.063	1.061	1.060	1.059	1.059	1.058	1.056	1.055	1.054	1.052	3
1.065	1.064	1.063	1.062	1.060	1.059	1.058	1.058	1.057	1.055	1.054	1.053	1.051	4
1.063	1.063	1.062	1.061	1.059	1.058	1.057	1.057	1.055	1.054	1.053	1.052	1.051	5
1.062	1.061	1.061	1.059	1.058	1.057	1.055	1.055	1.054	1.053	1.052	1.051	1.050	6
1.061	1.060	1.060	1.058	1.057	1.056	1.054	1.054	1.053	1.052	1.051	1.050	1.049	7
1.060	1.059	1.059	1.057	1.056	1.055	1.053	1.053	1.052	1.051	1.050	1.049	1.048	8
1.059	1.058	1.058	1.056	1.055	1.054	1.052	1.052	1.051	1.050	1.049	1.048	1.047	9
1.058	1.057	1.056	1.055	1.054	1.053	1.051	1.051	1.050	1.049	1.048	1.047	1.046	10
1.057	1.056	1.055	1.054	1.053	1.052	1.050	1.050	1.049	1.048	1.047	1.046	1.045	11
1.056	1.055	1.054	1.053	1.052	1.051	1.049	1.049	1.048	1.047	1.046	1.045	1.044	12
1.054	1.054	1.053	1.052	1.051	1.050	1.048	1.048	1.047	1.046	1.045	1.044	1.043	13
1.053	1.053	1.052	1.051	1.050	1.049	1.047	1.047	1.046	1.045	1.044	1.043	1.042	14
1.052	1.051	1.051	1.050	1.048	1.047	1.046	1.046	1.045	1.044	1.043	1.042	1.042	15
1.051	1.050	1.050	1.048	1.047	1.046	1.045	1.045	1.044	1.043	1.043	1.041	1.041	16
1.050	1.049	1.048	1.047	1.046	1.045	1.044	1.044	1.043	1.042	1.042	1.041	1.040	17
1.049	1.048	1.047	1.046	1.045	1.044	1.043	1.043	1.042	1.041	1.041	1.040	1.039	18
1.047	1.047	1.046	1.045	1.044	1.043	1.042	1.042	1.041	1.040	1.040	1.039	1.038	19
1.046	1.046	1.045	1.044	1.043	1.042	1.041	1.041	1.040	1.039	1.039	1.038	1.037	20
1.045	1.045	1.044	1.043	1.042	1.041	1.040	1.040	1.039	1.038	1.038	1.037	1.036	21
1.044	1.044	1.043	1.042	1.041	1.040	1.039	1.039	1.038	1.037	1.036	1.036	1.035	22
1.043	1.043	1.042	1.041	1.040	1.039	1.038	1.038	1.037	1.036	1.035	1.035	1.034	23
1.042	1.042	1.041	1.040	1.039	1.038	1.037	1.037	1.036	1.035	1.034	1.034	1.033	24
1.041	1.040	1.040	1.039	1.038	1.037	1.037	1.036	1.035	1.035	1.034	1.033	1.033	25
1.039	1.039	1.038	1.037	1.037	1.036	1.036	1.035	1.034	1.034	1.033	1.032	1.032	26
1.038	1.038	1.037	1.036	1.036	1.035	1.035	1.034	1.033	1.033	1.032	1.031	1.031	27
1.037	1.037	1.036	1.035	1.035	1.034	1.034	1.033	1.032	1.032	1.031	1.030	1.030	28
1.036	1.036	1.035	1.034	1.034	1.033	1.033	1.032	1.031	1.031	1.030	1.029	1.029	29
1.035	1.035	1.034	1.033	1.033	1.032	1.032	1.031	1.030	1.030	1.029	1.028	1.028	30
1.034	1.034	1.033	1.032	1.031	1.031	1.031	1.030	1.029	1.029	1.028	1.028	1.027	31
1.033	1.033	1.032	1.031	1.030	1.030	1.030	1.029	1.028	1.028	1.027	1.027	1.026	32
1.032	1.031	1.031	1.030	1.029	1.029	1.029	1.028	1.027	1.027	1.026	1.026	1.025	33
1.031	1.030	1.030	1.029	1.028	1.028	1.028	1.027	1.026	1.026	1.025	1.025	1.024	34
1.029	1.029	1.029	1.028	1.027	1.027	1.026	1.026	1.025	1.025	1.024	1.024	1.023	35
1.028	1.028	1.028	1.027	1.026	1.025	1.025	1.025	1.024	1.024	1.023	1.023	1.023	36
1.027	1.027	1.026	1.026	1.025	1.024	1.024	1.024	1.023	1.023	1.022	1.022	1.022	37
1.026	1.025	1.025	1.025	1.024	1.023	1.023	1.023	1.022	1.022	1.021	1.021	1.021	38
1.025	1.024	1.024	1.024	1.023	1.022	1.022	1.022	1.021	1.021	1.020	1.020	1.020	39
1.024	1.023	1.023	1.023	1.022	1.021	1.021	1.021	1.020	1.020	1.019	1.019	1.019	40
1.023	1.022	1.022	1.022	1.021	1.020	1.020	1.020	1.019	1.019	1.019	1.018	1.018	41
1.022	1.021	1.021	1.021	1.020	1.019	1.019	1.019	1.018	1.018	1.018	1.017	1.017	42
1.020	1.020	1.020	1.019	1.019	1.018	1.018	1.018	1.017	1.017	1.017	1.016	1.016	43
1.019	1.019	1.018	1.018	1.018	1.017	1.017	1.017	1.016	1.016	1.016	1.015	1.015	44
1.018	1.018	1.017	1.017	1.017	1.016	1.016	1.016	1.015	1.015	1.015	1.014	1.014	45
1.017	1.016	1.016	1.016	1.015	1.015	1.015	1.015	1.014	1.014	1.014	1.013	1.013	46
1.015	1.015	1.015	1.015	1.014	1.014	1.014	1.014	1.013	1.013	1.013	1.013	1.012	47
1.014	1.014	1.014	1.013	1.013	1.013	1.013	1.013	1.012	1.012	1.012	1.012	1.011	48
1.013	1.013	1.013	1.012	1.012	1.012	1.012	1.012	1.011	1.011	1.011	1.011	1.010	49

TABLE II.—VOLUME CORRECTION FACTORS (*Continued*).

Observed Temp., deg. F.	SPECIFIC GRAVITY AT 60 F./60 F.												
	0.498 to 0.502	0.503 to 0.507	Propane to 0.5079	0.508 to 0.512	0.513 to 0.517	0.518 to 0.522	0.523 to 0.527	0.528 to 0.532	0.533 to 0.537	0.538 to 0.542	0.543 to 0.547	0.548 to 0.552	0.553 to 0.557
	VOLUME CORRECTION FACTORS												
50	1.017	1.017	1.016	1.016	1.016	1.015	1.014	1.014	1.014	1.013	1.013	1.013	1.012
51	1.015	1.015	1.015	1.014	1.014	1.013	1.013	1.013	1.012	1.012	1.012	1.011	1.011
52	1.014	1.013	1.013	1.012	1.012	1.012	1.012	1.011	1.011	1.010	1.010	1.010	1.010
53	1.012	1.012	1.011	1.011	1.011	1.011	1.010	1.010	1.010	1.009	1.009	1.009	1.009
54	1.010	1.010	1.010	1.009	1.009	1.009	1.009	1.008	1.008	1.008	1.007	1.007	1.007
55	1.009	1.008	1.008	1.008	1.008	1.008	1.007	1.007	1.007	1.007	1.006	1.006	1.006
56	1.007	1.007	1.007	1.006	1.006	1.006	1.006	1.005	1.005	1.005	1.005	1.005	1.005
57	1.005	1.005	1.005	1.005	1.005	1.005	1.004	1.004	1.004	1.004	1.004	1.004	1.004
58	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.002	1.002	1.002
59	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.001	1.001	1.001	1.001	1.001	1.001
60	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
61	0.998	0.998	0.998	0.998	0.998	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999
62	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997
63	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.996	0.996	0.996	0.996	0.996	0.996
64	0.993	0.993	0.993	0.994	0.994	0.994	0.994	0.994	0.994	0.994	0.995	0.995	0.995
65	0.991	0.992	0.992	0.992	0.992	0.992	0.992	0.993	0.993	0.993	0.993	0.993	0.994
66	0.990	0.990	0.990	0.990	0.990	0.990	0.991	0.991	0.991	0.992	0.992	0.992	0.992
67	0.988	0.988	0.988	0.989	0.989	0.989	0.989	0.990	0.990	0.990	0.991	0.991	0.991
68	0.986	0.986	0.986	0.987	0.987	0.987	0.988	0.988	0.989	0.989	0.989	0.989	0.990
69	0.985	0.985	0.985	0.985	0.986	0.986	0.986	0.987	0.987	0.987	0.988	0.988	0.989
70	0.983	0.983	0.983	0.984	0.984	0.984	0.985	0.985	0.986	0.986	0.986	0.987	0.987
71	0.981	0.982	0.982	0.982	0.982	0.983	0.983	0.984	0.984	0.984	0.985	0.986	0.986
72	0.979	0.980	0.980	0.981	0.981	0.981	0.982	0.982	0.983	0.983	0.984	0.984	0.985
73	0.978	0.978	0.978	0.979	0.979	0.980	0.980	0.981	0.981	0.982	0.982	0.983	0.983
74	0.976	0.976	0.976	0.977	0.978	0.978	0.979	0.980	0.980	0.980	0.981	0.982	0.982
75	0.974	0.975	0.975	0.976	0.976	0.977	0.977	0.978	0.979	0.979	0.980	0.980	0.981
76	0.972	0.973	0.973	0.974	0.974	0.975	0.976	0.977	0.977	0.978	0.978	0.979	0.980
77	0.970	0.971	0.972	0.972	0.973	0.973	0.974	0.975	0.976	0.976	0.977	0.978	0.978
78	0.969	0.969	0.970	0.970	0.971	0.972	0.973	0.974	0.974	0.975	0.976	0.977	0.977
79	0.967	0.968	0.969	0.969	0.969	0.970	0.971	0.972	0.973	0.974	0.974	0.975	0.976
80	0.965	0.966	0.967	0.967	0.968	0.969	0.970	0.971	0.971	0.972	0.973	0.974	0.975
81	0.963	0.964	0.965	0.965	0.966	0.967	0.968	0.969	0.970	0.971	0.972	0.973	0.973
82	0.961	0.962	0.963	0.963	0.964	0.966	0.967	0.968	0.968	0.969	0.970	0.971	0.972
83	0.959	0.960	0.961	0.962	0.963	0.964	0.965	0.966	0.967	0.968	0.969	0.970	0.971
84	0.957	0.958	0.959	0.960	0.961	0.962	0.963	0.965	0.965	0.966	0.967	0.968	0.969
85	0.956	0.957	0.958	0.958	0.959	0.960	0.962	0.963	0.964	0.965	0.966	0.967	0.968
86	0.954	0.955	0.956	0.956	0.957	0.959	0.960	0.961	0.962	0.964	0.965	0.966	0.967
87	0.952	0.953	0.954	0.955	0.956	0.957	0.958	0.060	0.061	0.962	0.963	0.964	0.965
88	0.950	0.951	0.952	0.953	0.954	0.955	0.957	0.958	0.959	0.961	0.962	0.963	0.964
89	0.948	0.950	0.950	0.951	0.952	0.954	0.955	0.957	0.958	0.959	0.960	0.962	0.963
90	0.946	0.948	0.949	0.949	0.951	0.952	0.954	0.955	0.956	0.958	0.959	0.960	0.961
91	0.944	0.946	0.947	0.947	0.949	0.951	0.952	0.954	0.955	0.956	0.958	0.959	0.960
92	0.942	0.944	0.945	0.946	0.947	0.949	0.950	0.952	0.953	0.955	0.956	0.957	0.958
93	0.940	0.942	0.943	0.944	0.946	0.947	0.949	0.950	0.952	0.953	0.955	0.956	0.957
94	0.938	0.940	0.941	0.942	0.944	0.946	0.947	0.949	0.950	0.952	0.953	0.954	0.956
95	0.937	0.938	0.939	0.940	0.942	0.944	0.946	0.947	0.949	0.950	0.952	0.953	0.955
96	0.935	0.937	0.938	0.939	0.940	0.942	0.944	0.946	0.947	0.949	0.950	0.952	0.953
97	0.933	0.935	0.936	0.937	0.939	0.941	0.943	0.944	0.946	0.947	0.949	0.950	0.952
98	0.931	0.933	0.934	0.935	0.937	0.939	0.941	0.943	0.944	0.946	0.948	0.949	0.950
99	0.929	0.931	0.932	0.933	0.935	0.937	0.939	0.941	0.943	0.945	0.946	0.948	0.949
100	0.927	0.929	0.930	0.932	0.934	0.936	0.938	0.940	0.941	0.943	0.945	0.946	0.948
105	0.917	0.920	0.920	0.923	0.925	0.927	0.929	0.931	0.933	0.935	0.937	0.939	0.941
110	0.907	0.910	0.911	0.913	0.915	0.918	0.921	0.923	0.925	0.927	0.930	0.932	0.934
115	0.897	0.900	0.902	0.904	0.906	0.909	0.912	0.915	0.917	0.920	0.922	0.925	0.927
120	0.887	0.890	0.892	0.894	0.897	0.900	0.903	0.907	0.909	0.912	0.915	0.918	0.920
125	0.876	0.880	0.881	0.884	0.887	0.890	0.894	0.898	0.901	0.903	0.906	0.909	0.912
130	0.865	0.869	0.871	0.873	0.877	0.880	0.884	0.888	0.892	0.895	0.898	0.901	0.905
135	0.854	0.858	0.861	0.863	0.867	0.871	0.875	0.879	0.883	0.887	0.890	0.894	0.897
140	0.842	0.848	0.850	0.852	0.857	0.861	0.866	0.870	0.874	0.879	0.882	0.886	0.890

TABLE II.—VOLUME CORRECTION FACTORS (*Concluded*).

Specific Gravity at 60 F./60 F.													Observed Temp. deg. F
0.558 to 0.562	Iso-Butane to 0.5631	0.563 to 0.567	0.568 to 0.572	0.573 to 0.577	0.578 to 0.582	*n*-Butene to 0.5844	0.583 to 0.587	0.588 to 0.592	0.593 to 0.597	0.598 to 0.602	0.603 to 0.607	0.608 to 0.612	
Volume Correction Factors													
1.012	1.012	1.012	1.011	1.011	1.011	1.011	1.011	1.010	1.010	1.010	1.010	1.009	50
1.011	1.011	1.010	1.010	1.010	1.010	1.010	1.010	1.009	1.009	1.009	1.009	1.008	51
1.009	1.009	1.009	1.009	1.009	1.009	1.009	1.009	1.008	1.008	1.008	1.008	1.007	52
1.008	1.008	1.008	1.008	1.008	1.008	1.008	1.007	1.007	1.007	1.007	1.007	1.007	53
1.007	1.007	1.007	1.007	1.007	1.007	1.006	1.006	1.006	1.006	1.006	1.006	1.006	54
1.006	1.006	1.006	1.006	1.006	1.006	1.005	1.005	1.005	1.005	1.005	1.005	1.005	55
1.005	1.005	1.005	1.005	1.004	1.004	1.004	1.004	1.004	1.004	1.004	1.004	1.004	56
1.004	1.004	1.004	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	1.003	57
1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	1.002	58
1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	1.001	59
1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	60
0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	61
0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	62
0.996	0.996	0.996	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	63
0.995	0.995	0.995	0.995	0.995	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	64
0.994	0.994	0.994	0.994	0.994	0.994	0.995	0.995	0.995	0.995	0.995	0.995	0.995	65
0.993	0.993	0.993	0.993	0.993	0.993	0.993	0.993	0.993	0.994	0.994	0.994	0.994	66
0.991	0.991	0.992	0.992	0.992	0.992	0.992	0.992	0.992	0.993	0.993	0.993	0.993	67
0.990	0.990	0.990	0.990	0.991	0.991	0.991	0.991	0.991	0.992	0.992	0.992	0.992	68
0.989	0.989	0.989	0.989	0.990	0.990	0.990	0.990	0.990	0.991	0.991	0.991	0.991	69
0.988	0.988	0.988	0.988	0.988	0.989	0.989	0.989	0.989	0.990	0.990	0.990	0.990	70
0.986	0.987	0.987	0.987	0.987	0.988	0.988	0.988	0.988	0.989	0.989	0.989	0.989	71
0.985	0.986	0.986	0.986	0.986	0.987	0.987	0.987	0.987	0.988	0.988	0.988	0.988	72
0.984	0.984	0.984	0.985	0.985	0.986	0.986	0.986	0.986	0.986	0.987	0.987	0.987	73
0.983	0.983	0.983	0.984	0.984	0.985	0.985	0.985	0.985	0.985	0.986	0.986	0.986	74
0.981	0.982	0.982	0.983	0.983	0.983	0.984	0.984	0.984	0.984	0.985	0.985	0.986	75
0.980	0.981	0.981	0.981	0.982	0.982	0.982	0.983	0.983	0.983	0.984	0.984	0.985	76
0.979	0.979	0.980	0.980	0.981	0.981	0.981	0.981	0.982	0.982	0.983	0.983	0.984	77
0.978	0.978	0.978	0.979	0.979	0.980	0.980	0.980	0.981	0.981	0.982	0.982	0.983	78
0.977	0.977	0.977	0.978	0.978	0.979	0.979	0.979	0.980	0.980	0.981	0.981	0.982	79
0.975	0.976	0.976	0.977	0.977	0.978	0.978	0.978	0.979	0.979	0.980	0.980	0.981	80
0.974	0.975	0.975	0.975	0.976	0.977	0.977	0.977	0.978	0.978	0.979	0.979	0.980	81
0.972	0.973	0.974	0.974	0.975	0.976	0.976	0.976	0.977	0.977	0.978	0.978	0.979	82
0.971	0.972	0.972	0.973	0.974	0.975	0.975	0.975	0.976	0.976	0.977	0.977	0.978	83
0.970	0.971	0.971	0.972	0.973	0.974	0.974	0.974	0.975	0.975	0.976	0.976	0.977	84
0.969	0.970	0.970	0.971	0.971	0.972	0.973	0.973	0.974	0.974	0.975	0.975	0.976	85
0.967	0.968	0.968	0.969	0.970	0.971	0.971	0.972	0.972	0.973	0.973	0.974	0.975	86
0.966	0.967	0.967	0.968	0.969	0.970	0.970	0.971	0.971	0.972	0.972	0.973	0.974	87
0.965	0.966	0.966	0.967	0.968	0.969	0.969	0.970	0.970	0.971	0.971	0.972	0.973	88
0.964	0.964	0.965	0.966	0.967	0.968	0.968	0.969	0.969	0.970	0.970	0.971	0.972	89
0.962	0.963	0.963	0.964	0.966	0.967	0.967	0.967	0.968	0.969	0.969	9.970	0.971	90
0.961	0.962	0.962	0.963	0.964	0.965	0.966	0.966	0.967	0.968	0.968	0.969	0.970	91
0.959	0.960	0.961	0.962	0.963	0.964	0.965	0.965	0.966	0.967	0.967	0.968	0.969	92
0.958	0.959	0.960	0.961	0.962	0.963	0.964	0.964	0.965	0.966	0.966	0.967	0.968	93
0.957	0.958	0.958	0.959	0.961	0.962	0.962	0.963	0.964	0.965	0.965	0.966	0.967	94
0.956	0.957	0.957	0.958	0.959	0.961	0.961	0.962	0.963	0.964	0.964	0.965	0.966	95
0.954	0.955	0.956	0.957	0.958	0.959	0.960	0.960	0.961	0.962	0.963	0.964	0.965	96
0.953	0.954	0.954	0.956	0.957	0.958	0.959	0.959	0.960	0.961	0.962	0.963	0.964	97
0.952	0.953	0.953	0.954	0.956	0.957	0.957	0.958	0.959	0.960	0.961	0.962	0.963	98
0.950	0.951	0.952	0.953	0.955	0.956	0.956	0.957	0.958	0.959	0.960	0.961	0.962	99
0.949	0.950	0.951	0.952	0.953	0.954	0.955	0.956	0.957	0.958	0.959	0.960	0.961	100
0.943	0.943	0.944	0.946	0.947	0.949	0.949	0.950	0.951	0.953	0.954	0.955	...	105
0.936	0.937	0.938	0.939	0.941	0.943	0.944	0.945	0.946	0.948	0.949	0.950	...	110
0.930	0.930	0.931	0.933	0.935	0.937	0.938	0.939	0.940	0.942	0.944	0.945	...	115
0.923	0.924	0.925	0.927	0.929	0.931	0.932	0.933	0.934	0.936	0.938	0.940	...	120
0.916	0.916	0.918	0.920	0.923	0.925	0.927	0.927	0.928	0.931	0.933	0.934	...	125
0.908	0.909	0.911	0.913	0.916	0.918	0.921	0.921	0.923	0.925	0.927	0.929	...	130
0.901	0.902	0.904	0.907	0.909	0.912	0.914	0.914	0.916	0.919	0.921	0.923	...	135
0.893	0.895	0.896	0.900	0.903	0.905	0.907	0.908	0.910	0.913	0.915	0.918	...	140

APPENDIX VI

OPTIONAL USE OF DETONATION METER FOR MOTOR AND RESEARCH METHODS[1]

The Detonation Meter has been approved as optional equipment for the bouncing pin in the *Motor* (D 357 - 48)[2] and *Research* (D 908 - 48 T)[2] Methods. The meter is to be used with the present Weston Knockmeter to measure knock intensity and to make sure that fuel samples are being rated at Standard Knock Intensity as defined by the appropriate guide table. A Summary Report on the Investigation of Detonation Meter, Model 501,[3] contains the supporting data from cooperative tests which served as the basis for this recommendation.

APPARATUS

The Detonation Meter, Model 501, is shown in Fig. 1. It is available from the Phillips Petroleum Co., Chemical Products Department, Bartlesville, Oklahoma. A Detonation Pick-up, Type D-1, $\frac{7}{8}$ in. in diameter with 18 threads per inch, is required and may be purchased with the meter. A constant voltage transformer, such as Sola No. 30805, may be used and is necessary if the line voltage fluctuates more than plus or minus 5 v. This transformer may be purchased with the meter or from electrical supply houses.

INSTALLATION

Install the Lane-Wells pick-up in the bouncing pin hole with a torque of approximately 30 lb-ft. Use a new gasket each time the pick-up is installed. Connect the shielded input cable to the pick-up and to the terminal marked INPUT on the Detonation Meter. Disconnect the leads normally used with the Weston Knockmeter at the knockmeter terminals. The Detonation Meter is connected directly to the Weston Knockmeter, *and not to the Heater Element,* with the two-wire cable, as follows:

> **Caution: Because of dangerous voltages to ground make the connection to the Weston knockmeter before plugging the cable into the outlet marked METER on the instrument. To avoid electrical shock, the power switch of the detonation meter should be in the OFF position when attaching or detaching the plug. After installation it is advisable to shield the Weston knockmeter terminals.**

---

[1] This has been accepted by the Society. It is published as part of the 1948 Appendix to the ASTM Manual of Engine Test Methods for Rating Fuels.

[2] 1948 ASTM Manual of Engine Test Methods for Rating Fuels.

[3] See p. 340.

Connect the spade lugs of the two-wire cable to the terminals of the Weston Knockmeter, making certain that the positive lug, marked with a red dot, is connected to the positive terminal. Insert the plug of the two-wire cable in the socket labeled METER on the back of the instrument. Fasten the operational switch in any convenient place on the engine panel. Connect the lead from the operational switch to the socket marked SWITCH on the Detonation Meter and snap the switch to the OFF position. Then connect the Meter to the 110 v. a-c line. If this line voltage varies more than plus or minus 5 v., the constant voltage transformer must be used. Snap the power switch of the meter to ON and start the engine, allowing the pick-up and meter to warm up for at least 30 min.

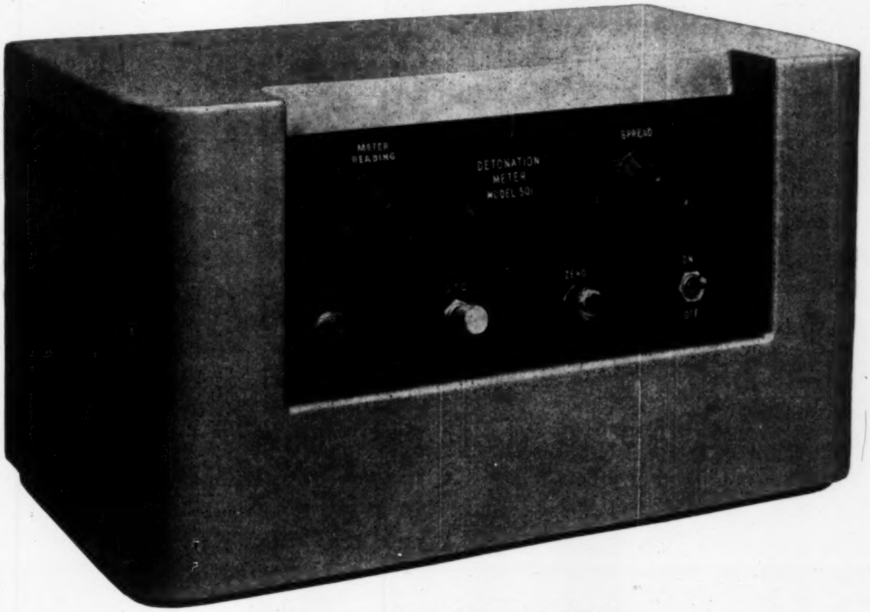


FIG. 1.—DETONATION METER.

ADJUSTING DETONATION METER

The Detonation Meter has three adjustable elements labeled ZERO, METER READING (marked SENSITIVITY on earlier meters), and SPREAD which are adjusted as follows:

(a) **Zero Adjustment.**—The zero adjustment of the meter must be checked when changing or installing pick-up units. Otherwise it need only be checked occasionally. Before making this check, warm up the meter for about 30 min. Then snap the operational switch to the OFF position and observe whether the knockmeter reads zero. If not, remove the screw cap marked ZERO on the Detonation Meter and make the necessary adjustment with a small screwdriver to bring the knockmeter pointer to zero. If the Detonation Meter has been warmed up sufficiently it should not be necessary to change this adjustment over a long period of time, but it should be checked occasionally as follows: With the operational switch in the OFF position, press the push button labeled S.T.C. (short time constant) on the meter so that a click is heard. This push button has two positions, one for slow response used in normal operations, and the other for fast response. The time constant differs about 20 to 1, so it is not difficult to determine which position the switch is in. If the zero adjustment is correct, press the push button to restore slow response, and snap the operational switch to the ON position.

(b) **Adjustments for Meter Reading and Spread.**—The controls for meter reading and spread are made so that each control has both a coarse and a fine adjustment with a 20-deg. lag between the two. Whenever the direction of rotation on the coarse adjustment is changed the control changes automatically to the fine adjustment. The operator can tell when he changes from the fine to the coarse adjustment by the increase in torque required to rotate the dial. It is not necessary to change these controls very much to cover the range 40 to 100 octane number, particularly if the initial setting of the meter is made at 85 octane number. However, this does not preclude making a setting at any desired octane level to follow the appropriate guide table, provided a check is obtained on the appropriate standardization fuel. It probably will be necessary to reset the meter for use above 100 octane number or below 40 octane number. With the engine operating under standard test conditions on a reference fuel, adjust the meter reading and spread as follows: With the dials marked METER READING and SPREAD both turned fully counter-clockwise, rotate the SPREAD dial approximately 15 deg. clockwise. Rotate the METER READING dial clockwise so that the knockmeter pointer is approximately mid-scale. Adjust the fuel-air ratio for maximum knock. If the maximum is not well defined, this is an indication that the sensitivity to change in octane number is too small. To increase the sensitivity rotate the SPREAD dial clockwise until the meter reads 100, then rotate the METER READING dial counter-clockwise until the meter is near mid-scale again. Change to the carburetor bowl containing the second reference fuel which differs from the first by not more than two octane numbers and determine its fuel-air ratio for maximum knock. The spread may then be determined by comparing the maximum readings for the two reference fuels. The desirable spread for two octane numbers is 15 to 30 divisions. In the low octane number range it may be necessary to use a lower spread but it never should be less than three knockmeter divisions per octane number (this also applies to the knockmeter and bouncing pin). If the spread is too small, increase the knockmeter reading approximately 20 divisions by rotating the SPREAD dial clockwise. Then rotate the METER READING dial counter-clockwise to restore the original reading. If the spread is not sufficient, repeat the procedure. Rotating the SPREAD dial clockwise and the METER READING dial counter-clockwise increases the spread.

To decrease the spread reverse this procedure. After the desired spread is obtained, the Detonation Meter is ready to use and a check should be made with an appropriate standardization fuel.

CHANGES IN MOTOR AND RESEARCH METHODS

For rating fuels the procedure is the same as presently specified for the Motor (D 357 – 47)[2] and Research (D 908 – 47 T)[2] methods except that the Detonation Meter is used instead of the bouncing pin. This will require the following changes in the Motor and Research methods and Supplement III on Operation in the 1948 ASTM Manual:

ASTM Designations.—Change the designation of the Motor Method to D 357 – 48, and that of the Research Method to D 908 – 48 T.

Section 3.—Add "or by a detonation meter and knockmeter" to the end of the second sentence.

Section 4.—Designate the present section as Paragraph (a) and add the following new Paragraph (b):

> (*b*) The Detonation Meter, Model 501,[a] shown in Fig. 3, may be used as optional equipment for the bouncing pin. A detonation pick-up, Type D-1, $\frac{7}{8}$ in. in diameter with 18 threads per inch, is required and may be purchased with the meter. A constant voltage transformer,[b] such as Sola No. 30805, may be used and is necessary if the line voltage fluctuates more than plus or minus 5 v.

New Section 11.—Add the above section, entitled "Adjusting Detonation Meter" as a new Section 11, renumbering the subsequent sections accordingly.

New Figure 3.—Add the accompanying Fig. 1 of the Detonation Meter as a new Fig. 3.

Section 11 (a).—In the second sentence, add "or with the detonation meter" after "bouncing pin." In the third sentence add "or the detonation meter adjustment" after "contact-point gap setting."

Section 11 (b).—In the first sentence add "or detonation meter" after "bouncing pin."

Tables I, III, IV and V.—Add "For Bouncing Pin Only" to the titles of these tables.

Tables II and VI.—Add a reference to Tables IA and VA in the title and headings.

Tables I and V.—To avoid frequent adjustment of the Detonation Meter, use guide Tables IA and VA which have been prepared for this meter, instead of the bouncing pin guide Tables I and V. This requires the addition of references to Table IA or VA in Section 6(*p*) of the Motor Method, Section 6(*o*) of the Research Method, Sections 8 and 11 (*b*) of both methods, and in Supplement III of the Manual in Sections 315 (*b*) and (*c*), and 319 (*c*).

---

[a] Available from the Phillips Petroleum Co., Chemical Products Department, Bartlesville, Okla.

[b] This transformer may be purchased with the meter or from electrical supply houses.

APPENDIX VII

PROPOSED METHOD OF TEST FOR SULFUR IN PETROLEUM OILS BY QUARTZ TUBE COMBUSTION[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method describes a procedure for the determination of sulfur, and for the determination of sulfur and chlorine in the presence of each other, in petroleum oils. The method is applicable to sulfur in any concentration and to chlorine in concentrations ranging from 0.01 to 3 per cent. For the determination of sulfur alone, it is particularly applicable to materials that cannot be analyzed directly by A.S.T.M. Method D 90, Test for Sulfur in Petroleum Products by the Lamp-Gravimetric Method.[2a] These include materials such as lubricating oils, fuel oils, gas oils, asphalts, and substances whose sulfur contents are above 3 per cent. For the co-determination of sulfur and chlorine, this method is particularly useful for the analysis of lubricating oils that have been blended with organic additives containing chlorine.

(*b*) The method gives erroneous sulfur results when applied to materials (for example, compounded lubricating oils) containing phosphorus and many metallo-organic compounds. Organic nitrogen compounds in the concentrations usually encountered in petroleum products do not interfere; however, nitrogen in concentrations higher than 1 per cent may cause slightly high sulfur values owing to formation of nitric acid during the combustion.

NOTE 1.—The determination of sulfur in the presence of such interfering substances should be made by means of A.S.T.M. method D 894, Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method.[3]

(*c*) The procedure for the determination of chlorine (Section 9) has been included primarily for correcting the sulfur content for the interference of chlorine. The results obtained may be slightly low owing to incomplete absorption of chlorine by the acidic absorbent used but, for chlorine contents of 3 per cent or less, are within the limits of reproducibility of the method. For highest accuracy this method should not be used for the determination of chlorine alone, even in concentrations less than 3 per cent. This limitation on the chlorine content does not affect the validity of the correction applied to the sulfur content, since the latter is a matter only of correcting the total acidity of the absorbent for acidity due to hydrogen chloride.

NOTE 2.—The determination of chlorine where the applicability of this method is in doubt should be made in accordance with A.S.T.M. Method D 808, Test for Chlorine in Lubricating Oils by Bomb Method.[3a]

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1948.

[2a] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[3] 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[3a] 1946 Book of A.S.T.M. Standards, Part III-A.

Outline of Method

2. The sample is vaporized and burned in a stream of air, and the oxidation completed by passing over quartz particles maintained at a temperature of 950 to 1000 C. The combustion products are passed through hydrogen peroxide which absorbs the sulfur as sulfuric acid and the chlorine as hydrochloric acid. The absorbent is then analyzed for total acidity and for chloride ion.

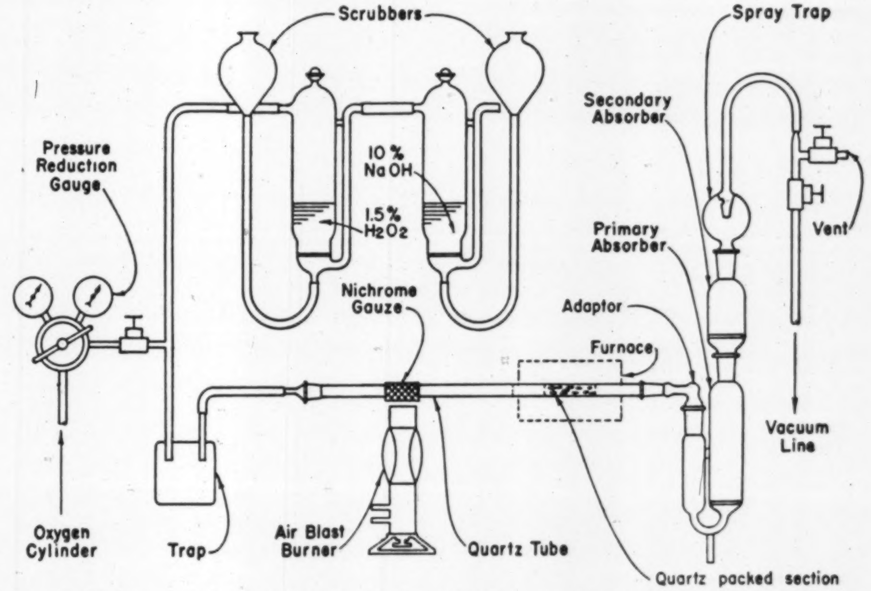

Scrubbers
Spray Trap
Secondary Absorber
Pressure Reduction Gauge
10 % NaOH
1.5 % H_2O_2
Vent
Primary Absorber
Nichrome Gauze
Adaptor
Furnace
Vacuum Line
Oxygen Cylinder
Trap
Air Blast Burner
Quartz Tube
Quartz packed section

FIG. 1.—Diagrammatic Sketch of Apparatus for Determination of Sulfur by Quartz Tube Combustion.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Combustion Tube*, made of transparent quartz as described in the Supplement, and assembled as shown in Fig. 1. The portion enclosed by dotted lines in Fig. 1 shall be heated by an electrical furnace which shall be capable of maintaining that portion of the tube at a temperature not lower than 950 C. The perforated quartz plate nearest the point of entry of the sample shall be flush with or not more than 10 mm. outside of the end of the furnace.

(*b*) *Absorbers and Spray Traps* as described in the Supplement.

(*c*) *Combustion Boat*, porcelain, 60-mm. in length for general use or 76 mm. in length for bulky samples.[4] The boat should easily enter the quartz combustion tube.

(*d*) *Combustion Atmosphere System*, as shown diagrammatically in Fig. 1. A precombustion furnace shall be included in the air line if the laboratory air service is likely to contain organic substances that will yield acids upon combustion. There shall also be included absorbers consisting of any suitable gas scrubbers capable of removing all acidic components from the combustion gas passing through at a rate of 3 liters per minute; one suitable arrangement of scrubbers is shown in Fig. 1. A source of oxygen at 5 psi. shall be provided to speed combustion of heavy carbonaceous residues. A vacuum pump capable of drawing air through the absorbers at a rate of 3 liters per minute per absorber shall also be provided.

---

[4] Coors Nos. 2 and 4 boats conform to these requirements.

(*e*) *Wire Gauze.*—A nichrome wire gauze "tunnel" made by bending a 60-mm. square of gauze into a U-shape of proper size to fit closely around the outside of the quartz tube.

(*f*) *Pyrometer.*—A pyrometer of suitable design, capable of indicating the temperature range from 950 to 1000 C.

(*g*) *Burner.*—A Fischer gas burner with improved head[5] (use of a blast-type burner may be expedient when inorganic residues are encountered).

(*h*) *Weighing Bottle.*—A Lunge weighing bottle.[6]

Reagents

4. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. References to water shall be understood to mean distilled water.

(*a*) *Ferric Alum Indicator Solution.*—Dissolve 350 g. of ferric ammonium sulfate crystals in 1 liter of water and add 200 ml. of 30 per cent aerated nitric acid.

(*b*) *Hydrogen Peroxide.*—Prepare by diluting 50 ml. of 30 per cent hydrogen peroxide to 1 liter with water. Store in a dark-colored glass-stoppered bottle.

(*c*) *Methyl Red Indicator Solution.*—Dissolve 0.2 g. of methyl red in 60 ml. of denatured alcohol and dilute to 100 ml. with water.

(*d*) *Nitric Acid.*—Dilute 415 ml. of concentrated nitric acid (sp. gr. 1.42) to 1 liter with water and aerate to remove oxides of nitrogen.

(*e*) *Nitrobenzene, technical grade.*—The nitrobenzene used shall not have more than a slight yellow color and shall not show a visible precipitate when tested with alcoholic silver nitrate solution.

(*f*) *Oxygen,* free of sulfur compounds, available at a pressure of 5 psi.

(*g*) *Silver Nitrate Solution* (*standard 0.1 N*).—To prepare 1 liter of solution, dissolve 17.0 g. of silver nitrate in water and dilute to volume. Store in a dark bottle. Standardize volumetrically against 0.23 to 0.24 g. of thoroughly dried sodium chloride.

(*h*) *Sodium Hydroxide Solution* (*standard 0.0624 N*).—To prepare 1 liter of solution, dissolve 2.5 g. of sodium hydroxide in water and dilute to volume. Store in a chemically resistant glass bottle. Removal of carbonate is not essential but care should be taken to minimize contamination from carbon dioxide in the air. Standardize by titration against standard acid in the absorber in the manner described in Section 8.

(*i*) *Sodium Hydroxide Solution* (*5 per cent*).—Prepare by dissolving 50 g. of sodium hydroxide pellets in 1 liter of water.

(*j*) *Thiocyanate Solution* (*standard 0.05 N*).—To prepare 1 liter of solution dissolve 4.85 g. of potassium thiocyanate or 3.80 g. of ammonium thiocyanate in water. To standardize, measure 40 ml. into a flask containing 5 ml. of 35 per cent nitric acid and 50 to 60 ml. of water. Swirling the flask constantly add 22 to 24 ml. of 0.1 *N* silver nitrate solution from a buret, then stopper and shake for 20 to 30 sec. Add 2 to 3 ml. of ferric alum indicator solution and back-titrate with thiocyanate solution to the red ferric thiocyanate end point. Calculate the normality from the total volumes of thiocyanate solution and silver nitrate solution used.

---

[5] This burner is available from the Fischer Scientific Co., Pittsburgh, Pa., Catalog No. 3-900.

[6] The Lunge weighing bottle available from Braun, Knecht, Heimann Co., San Francisco, Calif., Catalog No. 16731, is recommended.

Preparation of Apparatus

5. Assemble the apparatus as shown in Fig. 1. Introduce 30 ml. of the hydrogen peroxide solution into the primary (U-tube) absorber and 10 ml. into the secondary absorber above it. Adjust the vacuum control valve so that air is drawn through the absorber at a rate of about 3 liters per min. Heat the entire filled portion of the quartz tube so that a temperature of 950 to 1000 C. is maintained when air is drawn through the tube at the specified rate. Maintain this tube temperature and this rate of air flow throughout the determination.

Sampling

6. The optimum weight of sample to be used is one containing 35 to 50 mg. of sulfur. However, if the sulfur (and chlorine) content is less than 5 per cent, as is the case in most petroleum products, the sample size need be limited only to the capacity of the combustion boat. Three methods of sampling for analysis are suggested, as follows:

(*a*) For solids, heavy oils, or asphalts, weigh the sample directly in a porcelain combustion boat.

(*b*) For nonvolatile hygroscopic liquids and liquids that tend to creep and thus cannot be weighed directly in a boat, place the empty boat halfway into the quartz tube and, with air being drawn through the quartz tube at the required rate, add the desired amount of sample to the boat from a Lunge weighing bottle and immediately move the boat into the tube.

(*c*) To sample volatile liquids, use a 6-mm. diameter standard-wall Pyrex glass tube, 35 to 45 mm. in length, closed at one end and drawn to a fine capillary 35 to 45 mm. in length at the other. After weighing, fill the tube by warming the bulb in a small flame, inserting the tip of the capillary beneath the surface of the sample, cooling the tube with a small piece of solid carbon dioxide, and allowing the liquid to be drawn in. If a sufficient quantity is not drawn in, reheat the bulb to expel the sample, and repeat the process. Do not attempt to fill the bulb completely. Set the tube upright and cool by placing a piece of solid carbon dioxide against the bulb at a point just below the shoulder. Heat the capillary gently with a small flame to drive out any liquid remaining on the walls, then heat the lower end of the capillary and bend it downward so that it lies along the side of the bulb; do not seal the capillary. Wipe the tube and reweigh. Lay the tube in a porcelain combustion boat capillary side up and with the sealed end resting on a 4- to 5-mm. piece of 2-mm. glass rod placed in the boat. (This will cause the sample to be forced from the tube through expansion of the gas phase when it is heated.) Immediately move the boat into position in the quartz tube and proceed with the analysis.

Procedure for Combustion

7. (*a*) Break the connection between the air scrubbers and the quartz tube and insert the combustion boat and weighed sample a distance of 140 to 150 mm., pushing into position by means of a wire hook. Reconnect the air scrubbers and place the Fischer burner under the quartz tube at a point near the entrance end. Vaporize the sample at a slow, constant rate by gradually moving the burner toward the boat.

NOTE 3.—The original position of the burner with respect to the boat depends upon the volatility of the sample. For extremely volatile samples, the distance should be as great as possible. For less volatile materials the distance should be decreased, but in no case should the burner be placed directly under the boat at the start of the vaporization. Too rapid vaporization will be evidenced by pulsations in the visible vapor between the boat and the furnance. When thisoccurs, remove the burner immediately and allow the boat to cool for a few seconds, then proceed with the vaporization.

(*b*) After the sample has been vaporized from the boat, move the burner slowly along the tube to revaporize any material condensed between the boat and the furnace. Then place the burner directly beneath the boat, and place the nichrome wire gauze tunnel on the tube above the burner. Turn on the oxygen supply and adjust the flow until the air from the atmosphere just stops flowing through the scrubbers. Burn all residual carbon by moving the burner and gauze along the tube up to the furnace. If the sample was weighed in a capillary tube, heat the combustion tube to redness and cool several times in order to admit the oxygen necessary to burn out any residue.

(*c*) Turn off the oxygen supply and adjust the heat input to the furnace so that the latter will not overheat when the flow of gas through the tube is stopped.

(*d*) Disconnect the quartz tube from the adapter. Close the adapter opening with a glass stopper and allow a vacuum (50 to 60 cm. of mercury) to develop in the absorber. Close the vacuum control valve and slowly bleed air into the system through the air inlet valve, allowing the liquid to be drawn from the secondary absorber into the primary absorber. As the pressure slowly equalizes, remove the spray trap and rinse it with distilled water, collecting the washings in the secondary absorber. After all of the liquid has been drawn into the primary absorber, rinse the secondary absorber thoroughly, using a total of not more than 20 ml. of water. Remove the secondary absorber and wash the ground joint, both inside and out. Remove the stopper from the adapter and rinse the adapter and stopper with a small amount of water, collecting the washings in the primary absorber.

Procedure for Sulfate Determination

8. (*a*) Add 3 or 4 drops of methyl red indicator solution to the liquid in the absorber. Titrate the absorbent by introducing 0.0624 *N* sodium hydroxide solution from a buret into the smaller bulb of the absorber. Use a 10-ml. microburet if less than 10 mg. of sulfur is expected to be present in the absorber. Agitate during the titration by applying suction intermittently with the mouth to the top of the larger bulb (preferably by means of a piece of rubber tubing fitted to a rubber stopper of appropriate size).

NOTE 4.—When incomplete combustion of the sample occurs, the air drawn through the absorber has a characteristic taste or odor. In such cases, discard the determination.

(*b*) Once each day, make a blank determination on the hydrogen peroxide reagent.

Procedure for Chlorine Determination

9. (*a*) Quantitatively transfer the contents of the absorber after the titration in Section 8 to a 500-ml. glass-stoppered Erlenmeyer flask. Add approximately 2 ml. of the 5 per cent sodium hydroxide solution to the absorbent and heat at the boiling point until the indicator has been destroyed, adding an additional 10 ml. of the hydrogen peroxide solution if necessary. Cool, acidify with 10 ml. of the nitric acid, and add 10 ml. of nitrobenzene and 3 ml. of ferric alum indicator solution.

NOTE 5.—The chlorine determination may be omitted if the chlorine content of the sample is known to be negligible.

(*b*) Add 0.4 to 0.6 ml. of 0.05 *N* thiocyanate solution from a buret.

Swirling continuously, titrate with 0.1 N silver nitrate solution until the red ferric thiocyanate color is discharged; then add 2 to 5 ml. in excess. Stopper the flask tightly and shake vigorously to coagulate and remove the precipitate from the aqueous phase. Without refilling the buret, titrate with 0.05 N thiocyanate solution until a slowly fading red color is obtained. Stopper the flask, shake vigorously for 20 to 30 sec., and continue the titration to a red color that does not fade on further vigorous shaking.

NOTE 6.—Alternatively, the chlorine may be determined electrometrically.

Calculations

10. (*a*) Calculate the chlorine content of the sample, if determined, by means of the following equation:

Chlorine, per cent by weight =

$$\frac{(AN_1 - Bn) \times 3.55}{W}$$

where:

A = volume of standard silver nitrate solution used, in milliliters,

N_1 = normality of the standard silver nitrate solution,

B = total volume of standard thiocyanate solution used, in milliliters,

n = normality of standard thiocyanate solution, and

W = weight of sample, in grams.

(*b*) Calculate the sulfur content of the sample by means of the following equation:

Sulfur, per cent by weight =

$$\frac{(C - D) \times N_2 \times 1.60}{W} - 0.452X$$

where:

C = volume of standard sodium hydroxide solution, in milliliters, used in titrating the absorbent containing acidic combustion products,

D = volume of standard sodium hydroxide solution, in milliliters, used in titrating the hydrogen peroxide reagent blank,

N_2 = normality of the standard sodium hydroxide solution,

W = weight of sample, in grams, and

X = chlorine content, in per cent by weight.

Precision and Accuracy

11. (*a*) Results should not differ from the mean by more than the following amounts:

	Repeatability Same Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
Sulfur Content, per cent by weight:		
Under 3	0.02	0.03
3 or over	0.5 per cent of mean	1 per cent of mean
Chlorine Content, per cent by weight:		
Under 3	0.02	0.05

(*b*) The accuracy of the sulfur determination is of the same order of magnitude as the reproducibility. Chlorine results may be low by as much as 5 per cent of the amount present with substances whose chlorine contents are above 3 per cent.

(See Supplement, p. 338.)

SUPPLEMENT

APPARATUS

Combustion Train:

A1. The combustion train shall consist of the following:

(*a*) *Combustion Tube* of transparent fused quartz (Note 7) conforming to the dimensions shown in Fig. 2. An adapter as shown in Fig. 2 shall be provided for connection to the absorbers.

NOTE 7.—Transparent quartz tubes gradually become opaque, partly because of the nature of the samples burned, and to a smaller extent because of slow devitrification at the high temperature encountered during use. Opacity due to the former cause can be removed, and transparency restored, by etching the tube with a 1 per cent hydrofluoric acid solution for 24 hr., followed by careful heating with an oxygen-gas flame. If this treatment is not successful, the opaque section should be cut off and a new front-end section sealed in its place.

(*b*) *Absorber, Primary,*[7] of chemically resistant glass conforming to the dimensions shown in Fig. 2, provided with standard-taper glass joints for connection with the quartz tube and secondary absorber. A fritted disk with average pore diameter of 160 microns[8] shall be sealed in the larger of the two bulbs of the absorber. The fritted disk should be of such porosity that, when 50 ml. of water is placed in the absorber and air is passed through at the rate of 3.0 liters per minute in the forward direction, the pressure differential between the two sides of the absorber is between 15 and 23 cm. of water and the air is dispersed uniformly.

(*c*) *Absorber, Secondary, and Spray Trap* of chemically resistant glass conforming to the dimensions shown in Fig. 2, provided with standard-taper glass joints. The fritted disk shall have an average pore diameter of 14 microns.[9]

(*d*) *Furnace,* electric-resistance wound, or other type, as shown in Fig. 2, capable of maintaining the quartz-packed section and at least 1 in. beyond each end of this section at a temperature of 950 to 1000 C. The furnace dimensions and mounting shall be such that the perforated quartz plate nearest the point of entry of the sample can be flush with or not more than 10 mm. outside of the end of the furnace.

---

[7] This absorber is identical with the absorber described in A.S.T.M. Methods D 90.

[8] This porosity of fritted disk corresponds to Corning Extra Coarse."

[9] This porosity of fritted disk corresponds to Corning "Medium."

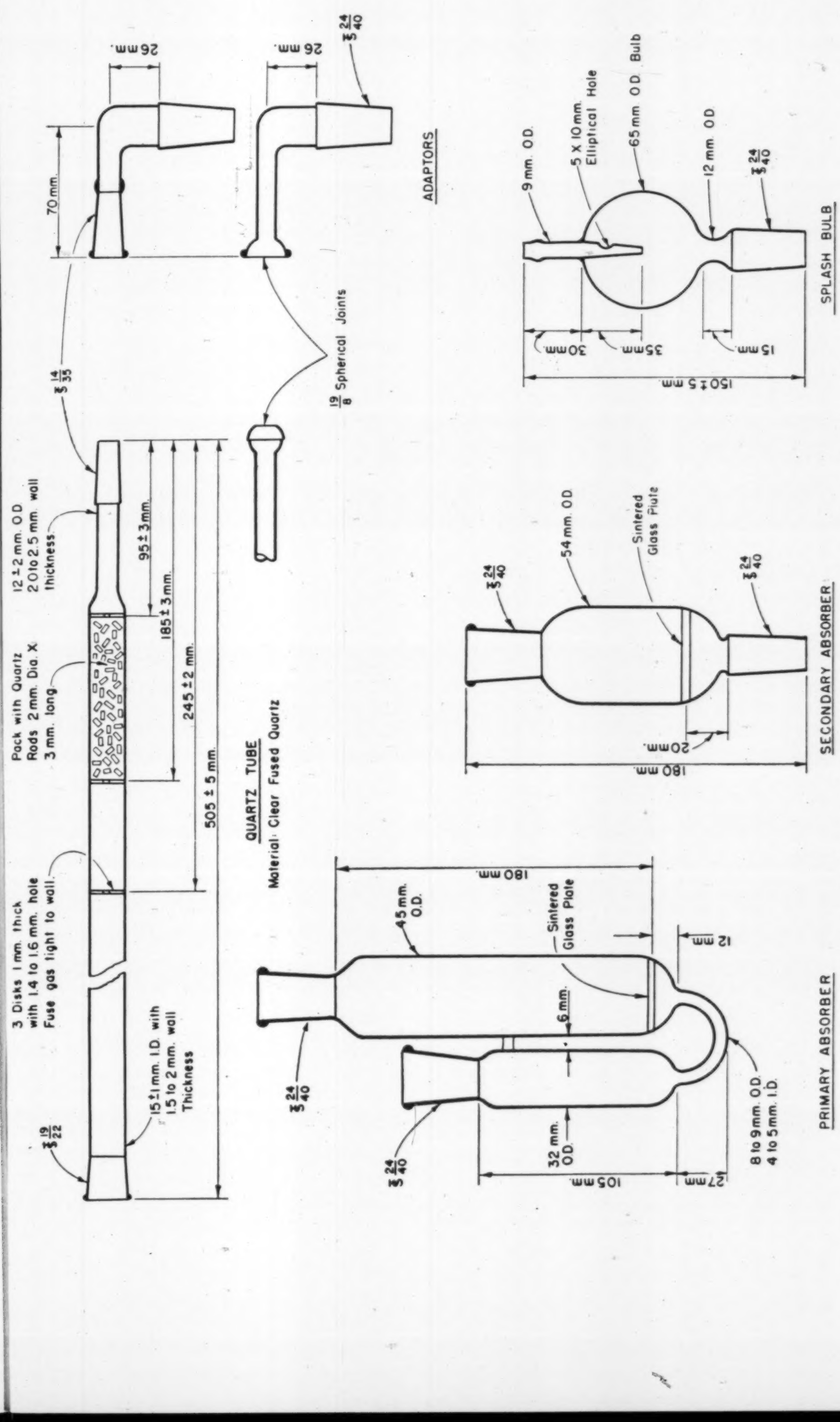

ADAPTORS
70 mm.
26 mm.
26 mm.
$\$ \frac{24}{40}$
$\$ \frac{14}{35}$
$\frac{19}{9}$ Spherical Joints
QUARTZ TUBE
Material · Clear Fused Quartz
12 ± 2 mm. O.D.
2.0 to 2.5 mm. wall thickness
95 ± 3 mm.
185 ± 3 mm.
245 ± 2 mm.
505 ± 5 mm.
Pack with Quartz Rods 2 mm. Dia. X 3 mm. long
3 Disks 1 mm. thick with 1.4 to 1.6 mm. hole Fuse gas tight to wall.
15 ± 1 mm. I.D. with 1.5 to 2 mm. wall Thickness
$\$ \frac{19}{22}$
SPLASH BULB
9 mm. O.D.
5 X 10 mm. Elliptical Hole
65 mm. O.D. Bulb
12 mm. O.D.
$\$ \frac{24}{40}$
30 mm.
35 mm.
15 mm.
150 ± 5 mm.
SECONDARY ABSORBER
$\$ \frac{24}{40}$
54 mm. O.D.
Sintered Glass Plate
$\$ \frac{24}{40}$
20 mm.
180 mm.
PRIMARY ABSORBER
180 mm.
45 mm. O.D.
Sintered Glass Plate
12 mm.
6 mm.
$\$ \frac{24}{40}$
$\$ \frac{24}{40}$
32 mm. O.D.
8 to 9 mm. O.D.
4 to 5 mm. I.D.
105 mm.
27 mm.

FIG. 2.—Detailed Drawing of Combustion and Absorption Apparatus for Determination of Sulfur by Quartz Tube Combustion.

APPENDIX VIII

SUMMARY REPORT ON INVESTIGATION OF THE DETONATION METER, MODEL 501

PREPARED BY SECTION III ON INSTRUMENTATION, OF THE DIVISION ON COMBUSTION CHARACTERISTICS, J. E. TAYLOR, CHAIRMAN.

Objective:

This investigation was made to evaluate the operating characteristics of Detonation Meter, Model 501, an instrument which was developed by the Phillips Petroleum Co. to replace the bouncing pin as a means of comparing knock intensities in the Motor (D 357 - 47) and Research (D 908 - 47 T) Methods of Test for knock characteristics of Motor Fuels. The primary objective was to determine whether the instrument is superior to the bouncing pin, with the underlying requirement that there be no significant change in the rating of a fuel from that obtained with the bouncing pin.

To fulfill the objective, significant number of comparative ratings had to be determined. In addition, such operating characteristics as stability, sensitivity, and performance factor had to be evaluated and guide curves established. Expression of operator reaction was a requisite because of the importance of this factor in reflecting the utility of the instrument as compared with the bouncing pin. It is believed all this has been accomplished in the test programs that have been completed.

Summary:

In February, 1946, investigation of Detonation Meter, Model 501, was assigned to the Engine and Accessories Modifications Group of the Motor Fuels Division, Cooperative Fuels Research Committee of the Coordinating Research Council, Inc. Since that time, eight individual investigations have been conducted to evaluate this instrument. Summary data from each of the investigations are incorporated in the Appendix to this report.

The first test program was exploratory in nature and was conducted mainly for the purpose of familiarization. An experimental model of the meter was circulated among seven laboratories and comparative performance data and fuel ratings with the bouncing pin were obtained by the Motor Method. Particular attention was given to operator acceptance which was good. Results of this program indicated that the detonation meter had definite possibilities and that continued investigation was warranted.

Consequently, ten production models of the meter were made available to the Knockmeter Panel, March 3, 1947. Subsequent investigation included test programs by both Motor and Research Methods, to compare performance characteristics of the meter and the bouncing pin, to establish tentative guide curves for the detonation meter, and to compare ratings on a wide variety of fuels with bouncing pin ratings. Results indicated that performance factor and stability are about the same as for the bouncing pin and that sensitivity is easily varied. In addition, operators were in unanimous agreement with the observation that "for the first time the operator can easily tell when the engine is at fault." The comparative ratings show that essentially the same ratings were obtained with the detonation meter as with the bouncing pin, except possibly when rating benzene standardization blends. For these blends, all of which contained 50 per cent benzene, the Research ratings tended to be somewhat lower with the detonation meter than with the bouncing pin. No issue was made of this result because the percentage of benzene in the blends was much higher than would be practical in commercial motor fuels.

On July 22, 1947, this activity was transferred from CFR to ASTM Committee D-2 on Petroleum Products and Lubricants and was immediately assigned to Section III, Instrumentation, of the Division on Combustion Characteristics. The first action of Section III was to recommend that additional meters be made available, particularly to members of the Motor Fuels Exchange Group, so that a significant number of comparative ratings for the detonation meter *versus* the bouncing pin could be obtained to supplement data already on record. In addition, the section agreed that an investigation should be made to determine the most suitable guide curve for each method, preferably a curve that would require only one setting of the meter. On October 30, 1947, the supplier advised that he was proceeding to manufacture 50 additional meters. These became available about January 15, 1948.

A program to establish the most suitable guide curves was carried out. The results show that curves developed with the detonation meter set to match the bouncing pin guide curve at 85 Octane Number for each method agree closely with the bouncing pin guide curves. These curves, covering a range from 40 to 100 Octane Number, were adopted by Section III on February 9, 1948. It was agreed that the guide curve tolerances permitted for the bouncing pin should be adhered to and that the barometric corrections used for the bouncing pin should be applied.

Using the guide curves recommended for the detonation meter, additional comparative ratings were obtained on diverse fuels under rigidly controlled operating conditions. The results furnished further evidence that there is no significant difference in ratings whether determined with the detonation meter or with the bouncing pin, the only exception being the blend containing a high percentage of benzene which again tended to rate lower with the detonation meter by the Research Method. Operators once more expressed strong approval of the meter.

To supplement the prescribed tests, all owners of detonation meters on record as of February 17, 1948, were requested to submit as many comparative ratings as they cared to make available to Section III. As a result, considerable data were compiled which included ratings for individual fuels which were determined with the detonation meter on one engine and with the bouncing pin on another engine. These ratings reflected not only instrumentation differences but also engine differences. The data were considerable and there was no indication that detonation meter ratings differed materially from bouncing pin ratings.

Comparative ratings reported to the Motor Fuels Exchange Group from November, 1947, through June, 1948, were reviewed. No marked differences between detonation meter ratings and bouncing pin ratings were found.

As the detonation meter, if adopted, would be required to operate under various altitude conditions, tests were made in the altitude chamber at the National Bureau of Standards to compare the relative performance of the detonation meter and the bouncing pin under simulated altitude conditions. These data show that the meter and the pin responded similarly to changes in barometric pressure.

Since some of the comparative results indicated that the detonation meter tended to underrate fuels containing high percentages of benzene by the Research Method, comparative tests were made with commercial type fuels containing practical quantities of benzene. These tests indicated that the meter has no effect on the rating of such fuels.

Reports of failures of the detonation pick-up unit used with the meter were brought to the attention of Section III during May, 1948. Apparently these failures were due to faulty construction and not to faulty design. To expedite solution of this problem, the Phillips Petroleum Co. has assumed responsibility for manufacture of the pick-up, and steps have been taken to rectify the difficulties. The pick-up, designated "Detonation Pick-Up, Type D-1," is available from the Phillips Petroleum Co.

All of the available comparative ratings for the detonation meter and bouncing pin are summarized in Table I. It should be emphasized that the differences shown are based on the individual ratings submitted by the participating laboratories. This method of analysis, which gives equal weight to each comparative rating submitted, was chosen because:

1. The number of test fuels or fuel types in each program varied.
2. The number of comparative ratings determined for each fuel in a particular program was not constant.
3. The number of comparative ratings obtained in each program differed materially.
4. Only individual comparative ratings were submitted for practically all fuels in Program 6 (Supplementary Comparative Ratings).

TABLE I.—SUMMARY OF AVERAGE DIFFERENCES BETWEEN OCTANE NUMBERS OBTAINED WITH THE DETONATION METER AND THE BOUNCING PIN.

INDIVIDUAL PROGRAMS

Program[a]	Date Reported	Number of Comparative Ratings		Average Difference Between Detonation Meter and Bouncing Pin Ratings (Based on Individual Ratings)			
				Arithmetic Difference (Disregarding Sign)		Algebraic Difference (Considering Sign) (D.M. minus B.P.)	
		Motor	Research	Motor	Research	Motor	Research
1	2- 5-47	50	..	0.60	....	+0.07	....
2A	10- 8-47	73	62	0.64	0.63	+0.32	−0.08
2B	10- 8-47	14	20	0.87	0.99	−0.57	−0.91
4A	6-18-48	71	66	0.22	0.27	+0.01	+0.07
4B	6-18-48	108	86	0.27	0.34	+0.03	−0.16
6	6-18-48	477	181	0.32	0.29	−0.01	+0.12
7	....	62	59	0.20	0.20	0.00	+0.01
8	....	3	9	0.27	0.11	+0.27	−0.01

AVERAGE DIFFERENCES FOR ALL AVAILABLE DATA

	Motor	Research
Total Number of Comparative Ratings	858	483
Average Difference (Based on Individual Ratings):		
(*a*) Arithmetic—Disregarding Sign	0.35	0.36
(*b*) Algebraic—Considering Sign (D. M. minus B.P.)	+0.02	−0.02

[a] Description of Programs:
- 1—Exploratory program using experimental detonation meter.
- 2—Test program on ten production model detonation meters:
  - A—Commercial type fuels
  - B—Benzene standardization blends
- 4—Additional comparative ratings using recommended guide curves:
  - A—Commercial type fuels.
  - B—Standardization blends (X-5, benzene, and leaded primary reference fuels).
- 6—Supplementary comparative ratings.
- 7—Exchange ratings, November, 1947, through June, 1948.
- 8—Blends of commercial motor fuels and benzene (obtained after Section III Meeting June, 18, 1948).

In reviewing these data, Section III concluded they are adequate to show that detonation meter ratings are comparable to those made with the bouncing pin.

Conclusions:

1. The test data obtained are adequate to show that the detonation meter does not change ratings significantly.

2. Performance characteristics (stability, sensitivity, performance factor) of the meter are comparable with those of the bouncing pin. The ease with which the sensitivity of the meter can be adjusted is very advantageous.

3. Altitude tests indicate that the detonation meter and the bouncing pin respond similarly to changes in barometric pressure.

4. In contrast to the bouncing pin, the detonation meter eliminates the factor of instrumentation in cases of faulty engine conditions.

5. Operator reaction to the detonation meter has been extremely favorable.

Recommendations:

1. That the Detonation Meter, Model 501, and Detonation Pick-Up, Type D-1 be adopted as optional equipment for the Motor and Research Methods.

2. That guide curves be used which are developed specifically for use with the detonation meter.

3. That bouncing pin barometric corrections be applied to the detonation meter guide curves.

4. That, for the present, the detonation meter and pick-up be available from the Phillips Petroleum Co.

Participants:

The following companies participated in this investigation:

1. Atlantic Refining Co., Philadelphia, Pa.
2. Esso Standard Oil Co., Baton Rouge, La.
3. Ethyl Corporation, Yonkers, N. Y.
4. Gulf Oil Corp., Philadelphia, Pa.
5. Gulf Research and Development Co., Pittsburgh, Pa.
6. National Bureau of Standards, Washington, D. C.
7. Phillips Petroleum Co., Bartlesville, Okla.
8. Standard Oil Development Co., Bayonne, N. J.
9. Standard Oil Development Co., Bayway, N. J.
10. Socony-Vacuum Oil Co., Brooklyn, N. Y.
11. Socony-Vacuum Oil Co., Paulsboro, N. J.
12. Sun Oil Co., Marcus Hook, Pa.
13. The Texas Company, Beacon, N. Y.
14. The Vickers Petroleum Co., Inc., Wichita, Kans.
15. Waukesha Motor Co., Waukesha, Wis.

DISCUSSION OF INVESTIGATIONS CONDUCTED ON DETONATION METER, MODEL 501

Following is a detailed discussion of each of the eight individual investigation programs that have been completed. Summary data for each program are included. In the several tables that present comparative ratings for the detonation meter *versus* the bouncing pin the results shown at the top of each table are based on average data, and those at the bottom on the individual ratings submitted by the participating laboratories. The latter were used to prepare Table I, wherein the reasons for analyzing the data in this manner are given.

1. Exploratory Program:

This program was prepared by the Knockmeter Panel of the CFR-MFD Engine and Accessories Modifications Group, August 21, 1946, with the following objectives in mind:

(*a*) To compare ratings obtained by using the detonation meter with bouncing pin ratings.

(*b*) To compare the detonation meter with the bouncing pin as to stability and sensitivity.

(*c*) To determine whether the detonation meter followed the bouncing pin guide curve.

(*d*) To determine operator reaction.

In this program an experimental model of the detonation meter was circulated among seven laboratories and investigation was limited to the Motor Method only. The results, summarized in Tables II, III and IV, were reported to the Engine and Accessories Modifications Group, February 5, 1947. It was also reported that operators reacted very favorably toward this experimental model of the meter.

TABLE III.—EXPLORATORY PROGRAM—PERFORMANCE CHARACTERISTICS, MOTOR METHOD.[a]

	Stability	Sensitivity	Performance Factor
Bouncing Pin[b]	0.91	7.2	0.12
Detonation Meter	1.24	13.4	0.06

[a] These data were obtained at the 70 octane number level using the bouncing pin test described on page 75 of the 1946 CRC Handbook.

[b] From a previous program of the Engine and Accessories Modifications Group on the shoulder suspended bouncing pin.

TABLE II.—INVESTIGATION OF DETONATION METER, MODEL 501, EXPLORATORY PROGRAM.
SUMMARY OF AVERAGE DATA

Fue	Comparative Octane Ratings by Motor Method				
	Detonation Meter	Bouncing Pin	D.M. minus B.P.	Average Deviation, plus or minus	
				D.M.	B.P
A—50% 1° Benzene + 50% *n*-heptane	40.5	41.6	−1.1	0.66	0.97
B—C-13 + 2.0 ml. TEL per gal.	84.0	83.9	+0.1	0.59	0.34
C—70% X-4 + 30% M-4	74.2	74.2	0.0	0.28	0.27
D—50% X-4 + 50% M-4 + 3.0 ml. TEL per gal	77.0	76.4	+0.6	0.46	0.44
E—X-4	92.4	91.8	+0.6	0.40	0.50
F—68.4% 1° Benzene + 31.6% C-13	85.7	86.0	−0.3	0.59	0.49
G—50% X-4, 35% F-6, 15% C-13 + 3.0 ml. TEL per gal.	96.2	95.2	+1.0	1.37	0.84
Average				0.62	0.55

SUMMARY OF INDIVIDUAL DATA

Total Number of Comparative Ratings	50
Average Difference (Based on Individual Ratings):	
(*a*) Arithmetic—Disregarding Sign	0.60
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	+0.07

These data show that there was no significant difference in ratings which ranged from 40 to 96 octane number, and that performance characteristics were comparable. The detonation meter did not follow the bouncing pin guide curve but this was not considered to be of any great import since few bouncing pin engine combinations will follow the curve prescribed for the Motor Method without adjustment of the bouncing pin.

2. Test Program on Ten Production Model Detonation Meters:

The program for testing these detonation meters by both Motor and Research Methods, was formulated March 28, 1947, for the following purposes:

TABLE IV.—EXPLORATORY PROGRAM—GUIDE CURVE DATA, MOTOR METHOD.

Octane Number	Micrometer Setting,[a] in.	
	Detonation Meter	Standard Bouncing Pin Values
40	0.706	0.738
50	0.660	0.686
60	0.600	0.620
70	0.526	0.533
75[b]	0.481	0.479
80	0.420	0.419
90	0.304	0.281
100	0.197	0.141

[a] Micrometer settings for standard knock intensity corrected to a barometric pressure of 29.92 in. of mercury. The engines were standardized on an X standardization fuel blend at 74.1 ± 0.2 octane number with bouncing pin.

[b] The detonation meter was adjusted to agree with the standard bouncing pin guide curve at the 75 octane number level. A spread of 15 ± 2 divisions per octane number was used.

(*a*) To establish tentative guide curves.
(*b*) To obtain comparative performance data.
(*c*) To obtain comparative ratings on a wide variety of fuels.

Results of this investigation, which were reported to Section III, Research Division I, October 8, 1947, are summarized in Tables V, VI, VII and VIII. It can be seen that performance factor and stability for the detonation meter were about the same as for the bouncing pin. The ease with which sensitivity could be varied when using the detonation meter received very favorable comment from the operators. Seven laboratories, each using their own detonation meters, reported data and the comparative ratings show that the meter had no marked effect on ratings. There was a tendency for the detonation meter to rate benzene standardization blends lower by the Research Method than did the bouncing pin but no particular concern was expressed over this result. These blends all contained 50 per cent benzene. The tentative guide curves for the meter were developed as the initial step in this program and were used throughout in determining the detonation meter ratings.

This program contributed additional evidence that the detonation meter was worthy of serious consideration. Section III recommended that additional meters be made available and that comparative ratings be obtained for Motor Fuel Exchange Group samples. More operators expressed strong approval of the meter. One paramount observation was "that for the first time the operator can easily tell when the engine is at fault." Ratings in this program covered the range from 40 octane number to *iso*octane plus 0.6 ml. of tetraethyllead per gallon.

TABLE V.—TEST PROGRAM ON TEN PRODUCTION MODEL DETONATION METERS.
TENTATIVE GUIDE CURVE DATA

Octane Number	Micrometer Setting,[a] in.			
	Motor Method		Research Method	
	Detonation Meter[b]	Standard Bouncing Pin Values	Detonation Meter[b]	Standard Bouncing Pin Values
40	0.700	0.738	0.567	0.580
45	0.682	0.713	0.552	0.562
50	0.658	0.686	0.537	0.542
55	0.631	0.655	0.517	0.521
60	0.599	0.620	0.498	0.499
65	0.563	0.580	0.473	0.474
70	0.525	0.533	0.450	0.447
75[c]	0.482	0.479	0.422	0.418
80	0.435	0.419	0.388	0.383
85	0.386	0.352	0.352	0.342
90	0.336	0.281	0.306	0.290
95	0.288	0.209	0.249	0.225
100	0.229	0.141	0.177	0.141
0.5[d]	0.157	....	0.098	....
1.5[d]	0.123	....	0.047	....
3.0[d]	0.103	....	0.009	....

[a] Micrometer settings for standard knock intensity corrected to a barometric pressure of 29.92 in. of mercury. Engines were standardized on X standardization fuel blends at 72.9 ± 0.3 octane number by the Motor Method and 74.8 ± 0.3 octane number by the Research Method with the bouncing pin.

[b] The detonation meter data are averages for the 15 and 30 spreads as there was little difference between them.

[c] Detonation meters were adjusted to agree with the standard bouncing pin guide curve at the 75 octane number level. A spread of 15 ± 2 divisions per 2 octane number was used in one case and 30 ± 2 divisions per 2 octane number in the other.

[d] Milliliters of tetraethyllead per gallon of *iso*octane.

TABLE VI.—TEST PROGRAM ON TEN PRODUCTION MODEL DETONATION METERS.
PERFORMANCE CHARACTERISTICS[a]

	Stability		Sensitivity		Performance Factor	
	Motor	Research	Motor	Research	Motor	Research
Bouncing Pin	0.59	0.96	7.3	7.8	0.08	0.21
Detonation Meter[b]	0.92	0.99	7.3	8.0	0.09	0.11
Detonation Meter[c]	1.10	1.22	14.6	15.5	0.08	0.26

[a] These data were obtained at the 73 octane number level for the Motor Method, and at the 75 octane number level for the Research Method. The bouncing pin test described on page 75 of the 1946 CRC Handbook, was used.

[b] Meter set for spread of 15 divisions per 2.0 octane number.

[c] Meter set for spread of 30 divisions per 2.0 octane number.

TABLE VII.—TEST PROGRAM ON TEN PRODUCTION MODEL DETONATION METERS.

COMMERCIAL TYPE FUELS

SUMMARY OF AVERAGE DATA

Fuel	Comparative Octane Ratings									
	Motor Method					Research Method				
	D.M.	B.P.	D.M. − B.P.	Average Deviation, plus or minus		D.M.	B.P.	D.M. − B.P.	Average Deviation, plus or minus	
				D.M.	B.P.				D.M.	B.P.
Stdz. 70% X-4 in *n*-heptane (Motor)	73.6	72.9	+0.7	0.34	0.16					
60 % X-4 in *n*-heptane (Research)						74.7	74.7	0.0	0.17	0.10
A—66⅔% Total Isobutylene Polymer + 33⅓% *Iso*octane	87.7	88.2	−0.5	0.59	0.74	0.45[a]	0.56[a]	−0.11[a]	0.05[a]	0.08[a]
B—Cracked Straight Run Blend + 3 ml. TEL per gal.	82.5	81.8	+0.7	0.42	0.38	92.1	91.9	+0.2	0.35	0.40
C—40% Catalytic Cracked + 40% Thermal + 20% Reformed Naphtha	74.6	74.1	+0.5	0.41	0.59	84.6	85.1	−0.5	0.56	0.38
D—40.7% Straight Run + 47.5% Thermal + 11.8% Vapor Recovery Gasoline	63.8	63.6	+0.2	1.02	0.90	67.8	68.2	−0.4	0.61	0.97
E—Straight Run	59.3	59.0	+0.3	0.94	0.80	60.1	60.6	−0.5	0.93	1.60
F—Alkylate Blending Agent and Aromatics + 4.6 ml. TEL per gal.	0.44[a]	0.46[a]	−0.02[a]	0.12[a]	0.16[a]	0.59[a]	0.55[a]	+0.04[a]	0.04[a]	0.04[a]
G—Straight Run + 0.65 ml. TEL per gal.	71.6	70.8	+0.8	0.60	0.60	71.7	71.7	0.0	0.44	0.59
H—Catalytic Cracked	78.6	78.5	+0.1	0.41	0.48	92.0	91.9	+0.1	0.31	0.25
J—Polyform + 3 ml. TEL per gal.	83.2	82.5	+0.7	0.55	0.66	93.6	93.2	+0.4	0.39	0.43
Average				0.59	0.59				0.47	0.59

SUMMARY OF INDIVIDUAL DATA

	Motor	Research
Total Number of Comparative Ratings	73	62
Average Difference (Based on Individual Ratings):		
(*a*) Arithmetic—Disregarding Sign	0.64	0.63
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	+0.32	−0.08

[a] Milliliters of tetraethyllead per gallon of *iso*octane—not included in computations.

TABLE VIII.—TEST PROGRAM ON TEN PRODUCTION MODEL DETONATION METERS.

BENZENE STANDARDIZATION BLENDS

SUMMARY OF AVERAGE DATA

Fuel[a]	Comparative Octane Ratings									
	Motor Method					Research Method				
	D.M.	B.P.	D.M.− B.P.	Average Deviation, plus or minus		D.M.	B.P.	D.M.− B.P.	Average Deviation, plus or minus	
				D.M.	B.P.				D.M.	B. P.
100% Fuel A	41.8	41.7	+0.1	0.45	0.51	51.2	52.0	−0.8	0.52	1.01
60% Fuel A, 40% Fuel B	....	....	....	....	....	74.5	75.5	−1.0	0.30	0.70
40% Fuel A, 60% Fuel B	78.7	80.1	−1.4	0.59	0.47	....	....	....	....	....
20% Fuel A, 80% Fuel B	....	....	....	....	....	95.1	96.2	−1.1	0.31	0.40
100% Fuel B	99.4	0.04[b]	....	0.67	0.04[b]	....	....	....	....	....
Average				0.57	0.49				0.38	0.70

SUMMARY OF INDIVIDUAL DATA

	Motor	Research
Total Number of Comparative Ratings	14	20
Average Difference (Based on Individual Ratings):		
(*a*) Arithmetic—Disregarding Sign	0.87	0.99
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	−0.57	−0.91

[a] Fuel A = 50 per cent benzene + 50 per cent *n*-heptane.
Fuel B = 50 per cent benzene + 50 per cent *iso*octane.

[b] Milliliters of tetraethyllead per gallon of *iso*octane — not included in computations.

3. *Program to Establish Most Desirable Guide Curve:*

The investigation to develop the most suitable guide curves for use with the detonation meter was drawn up November 20, 1947. In addition to developing a suitable guide curve for each method it was the desire to obtain a curve that would require only one setting of the meter to cover the entire octane number range. Seven laboratories participated in the program. After reviewing the data compiled in Table IX, Section III, adopted the curves in Figs. 1 and 2 on February 9, 1948. The data were obtained with the meter adjusted to agree with the standard guide curves for the bouncing pin at the level of 85 octane number for both methods. In preparing the guide curves the range was limited to 40 to 100 octane number.

TABLE IX.—PROGRAM TO ESTABLISH MOST DESIRABLE GUIDE CURVES.

Octane Number	Micrometer Settings in Inches for Standard Knock Intensity Corrected to a Barometric Pressure of 29.92 in. of Mercury									
	Motor Method					*Research Method*				
	Standard Bouncing Pin Values	Detonation Meter Adjusted to Agree with the Standard Bouncing Pin Guide Curve at the Following Octane Number Levels				Standard Bouncing Pin Values	Detonation Meter Adjusted to Agree with the Standard Bouncing Pin Guide Curve at the Following Octane Number Levels			
		85	90	95	100		85	90	95	100
40	0.738	0.708	0.701	0.672	0.636	0.580	0.555	0.538	0.531	0.503
45	0.713	0.683	0.683	0.644	0.613	0.562	0.539	0.524	0.517	0.490
50	0.686	0.658	0.656	0.621	0.589	0.542	0.521	0.508	0.502	0.473
55	0.655	0.628	0.619	0.592	0.564	0.521	0.505	0.491	0.485	0.454
60	0.620	0.596	0.589	0.565	0.535	0.499	0.486	0.470	0.465	0.439
65	0.580	0.560	0.555	0.529	0.501	0.474	0.463	0.447	0.443	0.415
70	0.533	0.521	0.516	0.491	0.465	0.447	0.438	0.423	0.418	0.394
75	0.479	0.473	0.469	0.446	0.422	0.418	0.410	0.397	0.392	0.368
80	0.419	0.424	0.414	0.393	0.364	0.383	0.379	0.367	0.363	0.340
85	0.352	0.353	0.348	0.328	0.302	0.342	0.343	0.331	0.325	0.305
90	0.281	0.284	0.281	0.261	0.238	0.290	0.299	0.290	0.283	0.265
95	0.209	0.224	0.226	0.208	0.183	0.225	0.243	0.234	0.225	0.212
100	0.141	0.184	0.181	0 171	0.142	0.141	0.165	0.158	0.152	0.140
0.5[a]	....	0.124	0.123	0.122	0.091	....	0.088	0.079	0.081	0.068
1.5[a]	....	0.092	0.089	0.084	0.060	....	0.040	0.031	0.029	0.021
3.0[a]	....	0.061	0.058	0.054	0.035	....	....	....	....	....

[a] Milliliters of tetraethyllead per gallon of *iso*octane.

The recommended guide curve for the Motor Method is shown in Fig. 1. From 75 to 90 octane number the curve for the detonation meter is practically identical with the standard curve for the bouncing pin but diverges from the latter at the higher octane number levels in one direction and at the lower octane number levels in the opposite direction.

The recommended guide curve for the Research Method is shown in Fig. 2. This curve agrees closely with the standard curve for the bouncing pin. The largest difference occurs at the low octane number levels. It was recommended by Section III that the guide curve tolerances permitted for the bouncing pin should be adhered to and that the barometric corrections used for the bouncing pin should apply.

4. *Additional Comparative Ratings Using Recommended Guide Curves:*

Table X is a summary of the results of a program to obtain additional comparative ratings of diverse fuels, covering the range 60 to 99 octane number and using the recommended guide curves for the detonation meter. Engine conditions were rigidly specified and this, plus

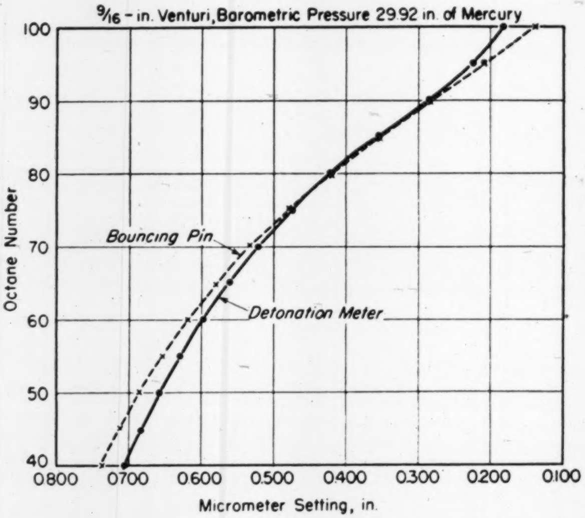


FIG. 1.—Recommended Detonation Meter Guide Curve, Motor Method.

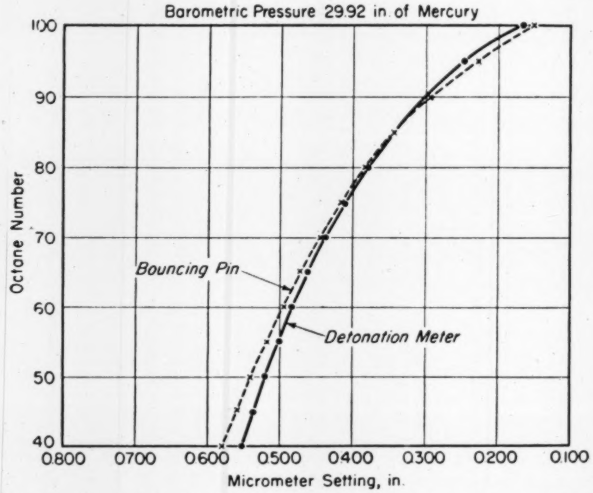


FIG. 2.—Recommended Detonation Meter Guide Curve, Research Method.

the added experience of operators in the use of the meter, probably accounts for the smaller differences between meter ratings and bouncing pin ratings than were found in the previous programs. This program was requested by letter February 5, 1948, and results were reported to Section III on June 18, 1948.

Twelve laboratories reported results obtained with their detonation meters. The only fuel of the thirteen included in the program to show any difference in rating that possibly warranted additional investigation was a blend of 68 per cent benzene and 32 per cent *n*-heptane. The detonation meter ratings for this blend tended to be somewhat lower than those for the bouncing pin by the Research Method. For the other twelve fuels there was no significant difference in ratings by either method. Widespread approval of the detonation meter by operators was reported.

TABLE X.—ADDITIONAL COMPARATIVE RATINGS USING RECOMMENDED GUIDE CURVES.
COMMERCIAL TYPE FUELS—SUMMARY OF AVERAGE DATA

Fuel	Motor Method					Research Method				
	D.M.	B.P.	D.M. minus B.P.	Average Deviation, plus or minus		D.M.	B.P.	D.M. minus B.P.	Average Deviation, plus or minus	
				D.M.	B.P.				D.M.	B.P.
K—Straight Run	60.2	60.2	0.0	0.57	0.51	61.9	61.7	+0.2	0.79	0.85
L—Straight Run + 3 ml. TEL per gal.	80.2	80.0	+0.2	0.38	0.26	81.3	81.2	+0.1	0.35	0.34
M—50% SR, 50% Catalytic Cracked	74.8	74.9	−0.1	0.43	0.43	82.7	82.8	−0.1	0.64	0.50
N—50% SR, 50% Catalytic Cracked + 3 ml. TEL per gal.	84.4	84.3	+0.1	0.36	0.34	93.3	93.1	+0.2	0.24	0.26
O—Catalytic Cracked	80.9	80.9	0.0	0.35	0.35	94.6	94.6	0.0	0.19	0.27
P—Catalytic Cracked + 3 ml. TEL per gal.	85.5	85.5	0.0	0.26	0.26	98.7	98.6	+0.1	0.30	0.48
Average				0.39	0.36				0.42	0.45
STANDARDIZATION FUELS—SUMMARY OF AVERAGE DATA										
Q—80% X-5, 20% *n*-Heptane	85.3	85.2	+0.1	0.22	0.17	97.8	97.8	0.0	0.23	0.15
R—65% X-5, 35% *n*-Heptane	74.2	74.1	+0.1	0.30	0.27	85.6	85.6	0.0	0.22	0.16
S—90% X-5, 10% *n*-Heptane	93.3	93.2	+0.1	0.33	0.21	....	....	...	....	....
T—50% X-5, 50% *n*-Heptane	59.7	59.5	+0.2	0.26	0.20	68.4	68.7	−0.3	0.28	0.37
U—60% *Iso*octane., 40% n-Heptane + 3 ml. TEL per gal.	81.3	81.2	+0.1	0.40	0.33	81.0	81.0	0.0	0.45	0.64
V—68% Benzene, 32% *n*-Heptane	64.6	65.0	−0.4	0.49	0.53	75.7	76.9	−1.2	0.87	0.91
Average				0.33	0.28				0.41	0.45

SUMMARY OF INDIVIDUAL DATA

	Commercial Type Fuels		Standardization Fuels	
	Motor	Research	Motor	Research
Total Number of Comparative Ratings	71	66	108	86
Average Difference (Based on Individual Ratings):				
(*a*) Arithmetic—Disregarding Sign	0.22	0.27	0.27	0.34
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	+0.01	+0.07	+0.03	−0.16

5. *Supplementary Comparative Ratings:*

On March 22, 1948, detonation meter owners on record as of February 17, 1948, were asked for any and all comparative ratings they could make available to Section III. A considerable number of ratings was reported. The ratings obtained were predominantly on commercial fuels although ratings on many other fuel types were included. In many cases the rating of a fuel was determined by the detonation meter on one engine and by the bouncing pin on another engine. These ratings, therefore, reflect not only instrumentation differences but also engine dif-

TABLE XI.—SUPPLEMENTARY COMPARATIVE OCTANE RATINGS.

SUMMARY OF AVERAGE DATA

Cooperators	Number of Comparative Ratings		Average Difference Between Detonation Meter and Bouncing Pin Ratings (Based on Individual Ratings)			
			Arithmetic Difference (Disregarding Sign)		Algebraic Difference (Considering Sign) (D.M. minus B.P.)	
	Motor	Research	Motor	Research	Motor	Research
Esso Standard Oil Co., La.	12	34	0.17	0.20	−0.08	+0.09
Ethyl Corp.	45	82	0.53	0.35	−0.26	+0.20
Gulf Oil Corp.	114	..	0.35	....	+0.04	....
Gulf Research and Development Co.	13	..	0.31	....	−0.23	....
Phillips Petroleum Co.	56	12	0.17	0.28	+0.02	−0.18
Standard Oil Development Co.:						
Bayonne	20	16	0.20	0.34	+0.11	+0.09
Bayway	44	24	0.28	0.25	+0.10	+0.10
Sun Oil Co.	117	13	0.18	0.23	−0.02	+0.08
Texas Co.	54	..	0.64	....	+0.03	....
Vickers Petroleum Co., Inc.	2	..	0.00	....	0.00	....

SUMMARY OF INDIVIDUAL DATA

	Motor	Research
Total Number of Comparative Ratings	477	181
Average Difference (Based on Individual Ratings):		
(*a*) Arithmetic—Disregarding Sign	0.32	0.29
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	−0.01	+0.12

TABLE XII.—NATIONAL MOTOR FUELS EXCHANGE GROUP.

COMPARATIVE OCTANE RATINGS

Date	Sample	No. of Ratings	Octane Number		D.M. minus B.P.	Average Deviation, plus or minus	
			D.M.	B.P.		D.M.	B.P.
SUMMARY OF AVERAGE DATA, MOTOR METHOD							
November, 1947	R-352	4	74.9	74.6	+0.3	0.18	0.28
December, 1947	R-353	5	78.5	78.4	+0.1	0.16	0.24
December, 1947	R-354	5	73.2	73.2	0.0	0.28	0.32
January, 1948	R-355	5	82.6	82.8	−0.2	0.26	0.26
February, 1948	R-356	7	84.2	84.2	0.0	0.29	0.19
March, 1948	R-357	7	78.8	79.1	−0.3	0.39	0.21
April, 1948	R-358	9	80.8	80.7	+0.1	0.29	0.27
May, 1948	R-359	6	57.5	57.3	+0.2	0.73	0.73
June, 1948	R-360A	7	64.3	64.2	+0.1	0.34	0.26
June, 1948	R-361A	7	77.9	77.8	+0.1	0.46	0.43
Average						0.34	0.32
SUMMARY OF AVERAGE DATA, RESEARCH METHOD							
November, 1947	R-352	2	78.3	78.3	0.0	0.60	0.50
December, 1947	R-353	4	84.3	84.3	0.0	0.40	0.18
December, 1947	R-354	4	76.0	76.1	−0.1	0.38	0.50
January, 1948	R-355	4	74.9	74.9	0.0	0.13	0.18
February, 1948	R-356	7	76.3	76.2	+0.1	0.33	0.26
March, 1948	R-357	9	86.0	86.1	−0.1	0.36	0.31
April, 1948	R-358	7	84.6	84.7	−0.1	0.14	0.17
May, 1948	R-359	6	58.6	58.7	−0.1	0.40	0.58
June, 1948	R-360A	8	67.4	67.2	+0.2	0.56	0.48
June, 1948	R-361A	8	81.5	81.3	+0.2	0.31	0.31
Average						0.36	0.35

SUMMARY OF INDIVIDUAL DATA

	Motor	Research
Total Number of Comparative Ratings	62	59
Average Difference (Based on Individual Ratings):		
(*a*) Arithmetic—Disregarding Sign	0.20	0.20
(*b*) Algebraic—Considering Sign (D.M. minus B.P.)	0.00	+0.01

ferences. No significant difference exists between detonation meter ratings and bouncing pin ratings. Table XI summarizes the results which were reported to Section III on June 18, 1948.

6. Exchange Ratings:

Compilation of comparative ratings reported to the Motor Fuels Exchange Group, from November, 1947, through June, 1948, illustrated once again that the detonation meter rated fuels the same as the bouncing pin. The ratings covered a range from approximately 60 to 85 octane numbers. The Exchange results are summarized in Table XII.

7. Barometric Corrections:

Tests were conducted in the altitude chamber at the Bureau of Standards during the week of May 2, 1948, to determine whether the response of the detonation meter was the same as that of the bouncing pin under altitude conditions. Pertinent results from this investigation are shown in Figs. 3 to 5.

Figure 3 shows the relation between barometric pressure and micrometer setting for constant knock intensity at three octane number levels. A Motor Method engine with a $\frac{3}{4}$-in. carburetor venturi was used for this work. Since the detonation meter and bouncing pin points are practically coincident at the four barometric pressures and the three octane levels, it follows that these two instruments behave similarly with regard to altitude corrections required to maintain a constant knock intensity. It seemed, therefore, that it should be proper to apply the corrections now specified in the method when the detonation meter is used.

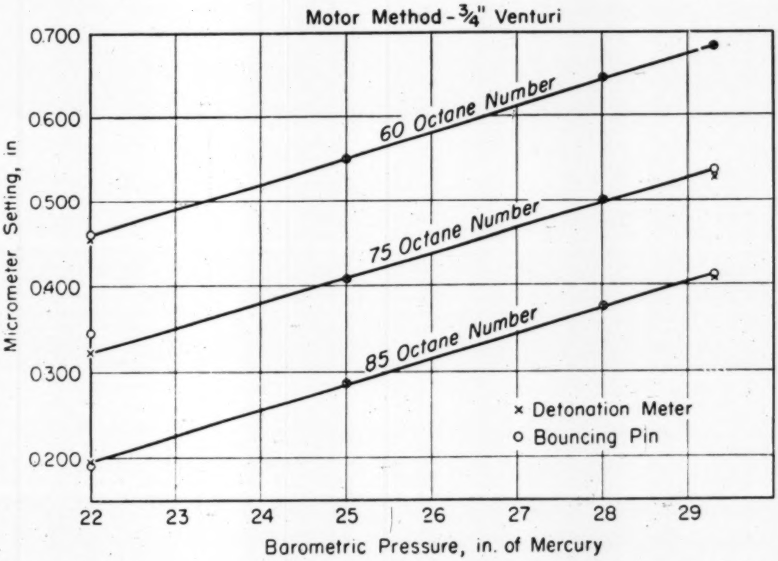


FIG. 3.—Barometric Pressure *versus* Micrometer Setting for Constant Knock Intensity.

Figure 4 shows the relation between the motor octane numbers obtained with the detonation meter and the bouncing pin for three blends of X-5 and *n*-heptane. These ratings were determined at four barometric pressures from 22.0 to 29.3 in. of mercury using a $\frac{9}{16}$-in. venturi in the carburetor. The ratings shown were estimated by relating the micrometer setting determined for

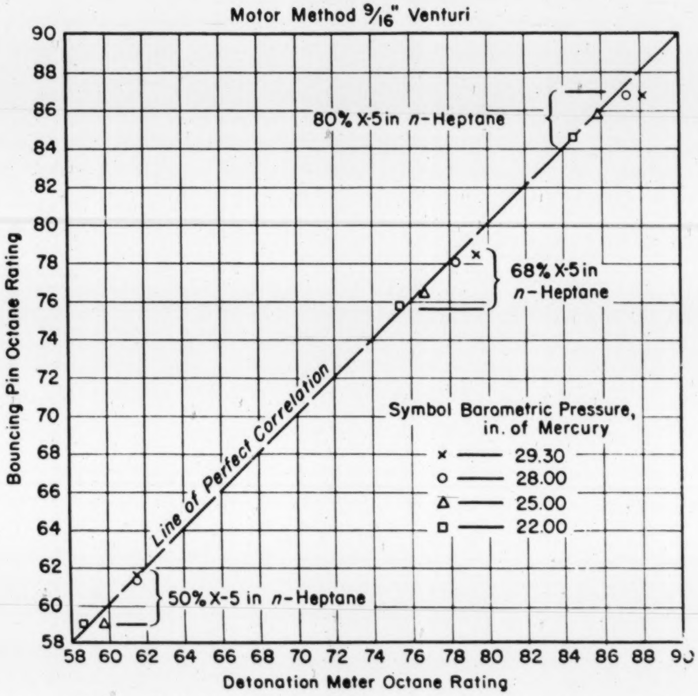


FIG. 4.—Relation Between Detonation Meter and Bouncing Pin Octane Ratings of Three X-5, *n*-Heptane Blends at Four Barometric Pressures.

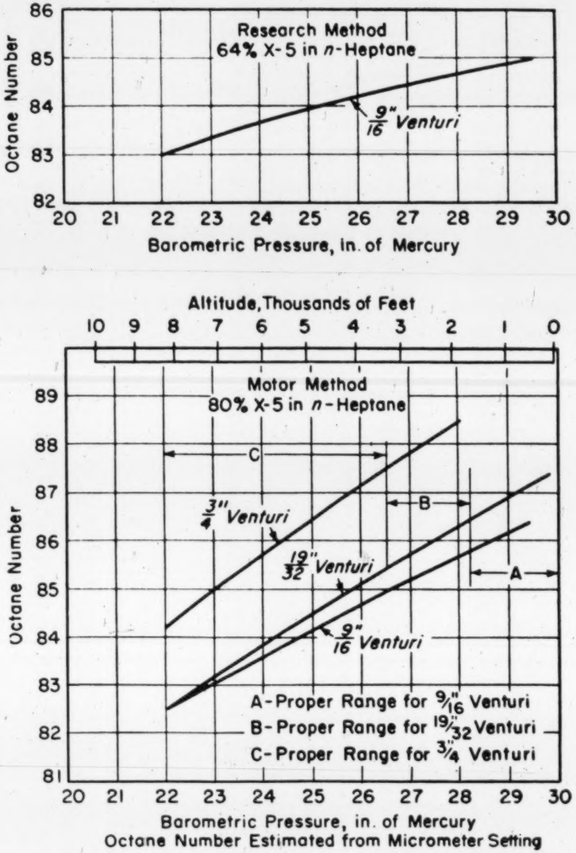


FIG. 5.—Effect of Barometric Pressure on the Octane Rating of X-5, *n*-Heptane Blends, Detonation Meter Ratings.

each X-blend to the appropriate guide curve. Since all of the points on the figure are very close to the line of perfect correlation, it was concluded that barometric pressure has no effect on the correlation between the bouncing pin and the detonation meter ratings of these fuels. Although the $\frac{9}{16}$-in. venturi is not expected to cover the wide range in barometric pressures covered by these tests, it was believed that comparative ratings outside the intended range of the venturi tended to increase the severity of the test and therefore magnified any differences in rating that existed.

TABLE XIII.—BLENDS OF COMMERCIAL MOTOR FUELS AND BENZENE.

Fuel	Comparative Octane Ratings					
	Motor			Research		
	Detonation Meter	Bouncing Pin	D.M. minus B.P.	Detonation Meter	Bouncing Pin	D.M. minus B.P.
100% Motor Fuel "A"	74.3	74.0	+0.3	74.8	74.8	0.0
90% Motor Fuel "A," 10% Benzene	75.4	75.2	+0.2	77.4	77.2	+0.2
80% Motor Fuel "A," 20% Benzene	77.0	76.7	+0.3	79.4	79.6	−0.2
80% Motor Fuel "B," 20% Benzene (Engine No. 1)	....	....	....	83.8	83.8	0.0
(Engine No. 2)	....	....	....	83.9	83.8	+0.1
80% Motor Fuel "C," 20% Benzene (Engine No. 1)	....	....	....	85.7	85.7	0.0
(Engine No. 2)	....	....	....	86.1	86.0	+0.1
80% Motor Fuel "D," 20% Benzene (Engine No. 1)	....	....	....	88.1	88.3	−0.2
(Engine No. 2)	....	....	....	88.0	88.2	−0.2

SUMMARY OF INDIVIDUAL DATA

	Motor	Research
Total Number of Comparative Ratings	3	9
Average Difference (Based on Individual Ratings):		
(a) Arithmetic—Disregarding Sign	0.27	0.11
(b) Algebraic—Considering Sign (D.M. minus B.P.)	+0.27	−0.01

Figure 5 shows the effect of barometric pressure on the Motor and Research ratings of blends of X-5 and *n*-heptane. These data indicate that changes in barometric pressure have a significant effect on the rating of these fuels by both the Motor and Research Methods and suggest that the present altitude corrections may not be adequate. It is of interest to note from the Motor Method curves that a practically constant octane rating for a definite blend is obtained with each venturi if the test is conducted at a barometric pressure in the middle of the proper range for that venturi. This problem is outside the scope of this report but is reported here as a matter of interest since it was discovered while conducting a test program on the detonation meter.

8. Blends of Commercial Motor Fuels and Benzene:

The data obtained in this program were requested May 28, 1948, and were reported after the meeting of Section III, June 18, 1948. The purpose of this program was to investigate motor fuels which contained what could be considered practical quantities of benzene. Previous data had shown that the detonation meter tended to rate benzene blends lower than did the bouncing pin by the Research Method. The blends which showed these differences contained from 50 to 68 per cent benzene which introduced a question as to their significance. The comparative detonation meter *versus* bouncing pin ratings that were determined are summarized in Table XIII. The results indicate that the meter has no effect on the rating of motor fuels containing conservative amounts of benzene.

APPENDIX IX

PROPOSED METHOD OF TEST FOR SODIUM IN LUBRICATING OILS AND LUBRICATING OIL ADDITIVES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes the zinc carbonate – triple acetate procedure for the determination of sodium in lubricating oil and lubricating oil additives containing from 0.001 to 2 per cent sodium. There is no interference from aluminum, calcium, barium, lead, iron, copper, cadmium, zinc, tin, chlorine, phosphorus, and sulfur, when these are present in amounts usually found in lubricating oils and additives. Small amounts of sulfate and moderate amounts of potassium do not interfere except when present simultaneously. Large amounts of phosphorus tend to interfere with the determination of small amounts of sodium because of incomplete removal of phosphorus by the zinc carbonate treatment.

NOTE 1.—The method is also applicable, but with somewhat decreased accuracy, to samples of higher sodium and phosphorus contents than are indicated in Table I relating sample size and concentration. For a general method of analysis for metals in lubricating oils, see A.S.T.M. Method D 811.[2]

Outline of Method

2. The sample is completely oxidized by dry ashing; the oil is allowed to burn freely until only a coke or tar remains, and the free carbon on the sides of the dish is ignited at a dull red heat (550 C.) until all carbonaceous matter has disappeared. The residue from the dry-ashing procedure is taken up in dilute hydrochloric acid and neutralized with dilute ammonium hydroxide. Phosphates are separated by the addition of a large excess of zinc carbonate to the neutral solution. The sodium is determined in the filtrate by precipitation with zinc uranyl acetate reagent and weighing as sodium zinc uranyl acetate.

TABLE I.—SIZE OF SAMPLE FOR VARYING SODIUM AND PHOSPHORUS CONTENTS.

Sodium Content, per cent by weight	Maximum Phosphorus Content, per cent by weight	Sample Weight, g.
0.001 to 0.02........	0.2	20 ± 0.1
0.020 to 0.20........	2.0	2.0 ± 0.01
0.20 to 2.0.........	20	0.2 ± 0.001

Apparatus

3. The following apparatus is required:

(*a*) *Platinum Dish*, shallow, of 50-ml. or 100-ml. capacity.

(*b*) *Muffle Furnace*, capable of operating at a variable temperature up to 600 C.

(*c*) *Filter Crucible*, porcelain of fine porosity.

Reagents

4. Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Com-

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1948.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

mittee on Analytical Reagents of the American Chemical Society; where such specifications do not exist, use the best available grade.

(*a*) *Ammonium Hydroxide Solution (12 per cent)*.—Prepare by mixing 495 ml. of concentrated ammonium hydroxide (sp. gr. 0.90) and 505 ml. of water.

(*b*) *Ammonium Nitrate Solution (1 per cent)*.—Prepare by dissolving 10 g. of ammonium nitrate (NH_4NO_3) in 1 liter of water.

(*c*) *Ethyl Ether (sp. gr. 0.712 to 0.714 at 25/25 C.)*

(*d*) *Hydrochloric Acid Solution (3 per cent)*.—Prepare by mixing 70 ml. of concentrated hydrochloric acid (sp. gr. 1.18) and 930 ml. of water.

(*e*) *Indicator Paper*.—Test paper for indicating pH values between 3 and 11.[3]

(*f*) *Sodium Zinc Uranyl Acetate Wash Solution*.—Prepare a saturated solution in absolute ethyl alcohol. Filter just before use.

(*g*) *Zinc Carbonate*.—$ZnCO_3$ powder containing less than 0.002 per cent sodium.

(*h*) *Zinc Uranyl Acetate Solution (saturated)*.—Dissolve 10 g. of uranium acetate dihydrate ($UO_2(C_2H_3O_2)_2 \cdot 2H_2O$) in 49 ml. of water containing 6 ml. of 30 per cent acetic acid solution. Dissolve 30 g. of zinc acetate dihydrate ($Zn(C_2H_3O_2)_2 \cdot 2H_2O$) in 32 ml. of water containing 3 ml. of 30 per cent acetic acid solution. Mix the two solutions and allow to stand for 24 hr. Filter to remove excess zinc uranyl acetate. Store the reagent in a chemically resistant glass-stoppered bottle.[4]

Procedure

5. (*a*) Into a 50 or 100-ml. platinum dish, weigh a well-mixed sample containing from 0.2 to 4 mg. of sodium as indicated in Table I.

NOTE 2.—Sodium may be determined with somewhat decreased accuracy in samples containing higher concentrations of sodium and phosphorus than are indicated in Table I by taking an appropriate aliquot of the sample solution prior to the separation of phosphate with zinc carbonate. In no case should the aliquot contain more than 8 mg. of sodium or 150 mg. of phosphate.

(*b*) Heat the dish with a bunsen burner until the contents ignite and burn readily, and move the dish and flaming contents to a hot plate. Maintain the hot plate at such a temperature that the sample continues to burn at a uniform and moderate rate, leaving only ash and carbon when burning ceases. Place the dish in a cool furnace (below 300 C.), gradually increase the temperature to 550 ± 50 C., and continue the ignition until the oxidation of carbon is complete.

(*c*) Cool, and add 50 ml. of distilled water and 1 to 2 ml. of concentrated hydrochloric acid. Heat to boiling and boil gently for 15 to 20 min. Remove the solution from the hot plate and add 12 per cent ammonium hydroxide solution until a precipitate begins to form. If no precipitate forms, adjust the solution to pH 7 as indicated by a small piece of indicator paper which has been added to the solution. Add 3 per cent hydrochloric acid solution until the precipitate just dissolves or until the solution is pH 5, as indicated by the indicator paper.

NOTE 3.—Close control of the acidity of the solution is necessary to avoid dissolving a larger quantity of zinc carbonate than is desirable in the subsequent separation of phosphates. The presence of a large amount of zinc in the solution increases the chance of spattering when the volume is reduced to 2 ml. prior to the precipitation of sodium.

(*d*) Add 5.0 g. of zinc carbonate, with constant stirring, and let stand at room temperature for at least 6 hr. or overnight. Add approximately 0.3 g. of ammonium nitrate, filter through a fine porosity filter paper, and wash five or six times with cold 1 per cent ammonium nitrate solution, catching the filtrate in a 100-ml. beaker. Evaporate the solution to a volume of approximately 2 ml.

---

[3] Alkacid or Hydrion papers, or their equivalent, are satisfactory for this purpose.

[4] A Pyrex bottle is satisfactory for this purpose.

(*e*) Add 25 ml. of freshly filtered zinc uranyl acetate reagent (Note 4), with constant stirring, to the 2-ml. solution. Allow the solution to stand for 15 to 30 min. and filter through a prepared porous porcelain filter crucible. Prepare the crucible by rinsing successively inside and out with distilled water, alcohol, and ether. Allow the crucible to stand 10 to 20 min. before weighing. Wash the sodium zinc uranyl acetate from the beaker with approximately 35 ml. of reagent. Wash the precipitate five or six times with freshly filtered saturated sodium zinc uranyl acetate alcoholic wash solution. Rinse three or four times with ether, removing the ether completely by suction. Wipe the outside of the crucible with a lint-free cloth, allow to stand at least 20 min., and weigh.

NOTE 4.—At least a ten-fold excess of the zinc uranyl acetate reagent must be used to precipitate the sodium quantitatively.

(*f*) Make a blank determination following the above procedure and using the same amounts of all the reagents, but omitting the sample.

NOTE 5.—If the weight obtained for the precipitate from the blank is more than 5 mg., the zinc carbonate should be repurified. This can be done most easily by extracting the zinc carbonate with hot distilled water, filtering on a sintered glass or porcelain funnel, and drying with alcohol and ether.

Calculation

6. Calculate the sodium content of the sample as follows:

$$\text{Sodium, per cent by weight} = \frac{(A - B)(1.495)}{W}$$

where:

A = weight of sodium zinc uranyl acetate precipitate, in grams,

B = weight of the blank precipitate, in grams, and

W = weight of the original sample, in grams.

Precision

7. Results should not differ from the mean by more than the following amounts:

Sodium Content, per cent by weight	**Repeatability** Same Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
0.001 to 0.02	0.0002	0.0004
0.02 to 0.2	0.002	0.004
0.2 to 2.0	0.02	0.04

APPENDIX X

PROPOSED METHOD OF MEASURING THE TEMPERATURE OF PETROLEUM AND PETROLEUM PRODUCTS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes the standard procedures for determining temperatures of petroleum and its products when in a liquid state.

Outline of Method

2. Temperatures of liquid petroleum products are determined at specified locations in tanks, ships and barges, tank cars and trucks, because accurate average temperatures and gage readings are necessary to calculate the volumes at the standard temperature of 60 F. For a non-pressure tank a desired temperature is obtained by lowering an A.S.T.M. tank thermometer of appropriate range through the gaging hatch to the specified level, and then withdrawing and reading the thermometer quickly after the entire thermometer assembly has attained the temperature of the product. This procedure is also used for low-pressure tanks equipped with gaging hatches on stand pipes, and for any pressure tank which has a pressure gaging lock. For pressure tanks equipped with thermometer wells, temperatures are obtained by reading thermometers placed in the wells with their bulbs at the desired tank levels. In all cases where more than one temperature is determined, the average temperature of the product is calculated arithmetically from the observed temperatures.

APPARATUS

Thermometers

3. Total immersion thermometers with etched graduated glass stems and with bulbs made of Corning normal or equivalent thermometric glass, mercury filled, with nitrogen gas above the mercury column, and con-

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1948.

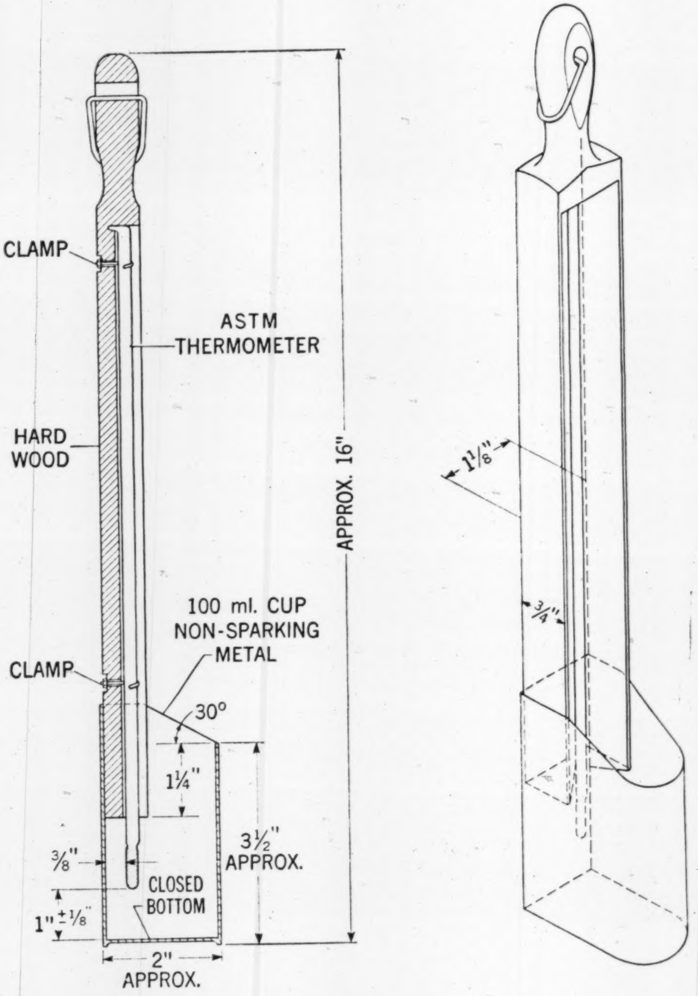

CLAMP
ASTM
THERMOMETER
HARD
WOOD
APPROX. 16"
100 ml. CUP
NON-SPARKING
METAL
CLAMP
30°
1¼"
3½"
APPROX.
⅜"
CLOSED
BOTTOM
1" ± ⅛"
2"
APPROX.
1⅛"
¾"

FIG. 1.—TYPICAL CUP CASE ASSEMBLY.

forming to the Tentative Specifications E 1 - 48 T[2] for the A.S.T.M. Thermometers listed in Table I shall be used.

TABLE I.—THERMOMETERS.

NAME	ASTM THERMOMETER	RANGE	LENGTH, IN.	GRADUATION	ACCURACY
ASTM Tank Low......	58 F - 48 T	−30 to +120 F.	12	1 F.	±0.5 F.
ASTM Tank Medium...	59 F - 48 T	0 to 180 F.	12	1 F.	±0.5 F.
ASTM Tank High......	60 F - 48 T	170 to 500 F.	12	2 F.	±1.0 F.
Angle Stem............	...	suitable range	12[a]	1 F.	±1.0 F.

[a] Length of graduated portion.

Thermometer Assemblies

4. (a) It is necessary to enclose thermometers in suitable cases to protect them during use. Three types of cases are recommended for the A.S.T.M. tank thermometers, the particular type used depending on the product and the type of tank.

(b) **Cup Case.**—This assembly is illustrated in Fig. 1. The cup case may be made of either varnished hardwood or non-sparking metal, with a cup of at least 100 ml. (6.1 cu. in.) capacity, and with dimensions such that the side of the bulb will be at least 3/8 in. from the nearest wall and the bottom of the bulb 1 ± 1/8 in. above the bottom of the cup.

(c) **Flushing Case.**—This assembly is illustrated in Fig. 2. The flushing case consists of a cylindrical chamber of at least 200 ml. (12.2 cu. in.) capacity and an armored or protecting tube rigidly attached to the chamber, both chamber and tube being made of non-sparking metal. The design and construction should be such that not more than 2 min. of flushing time will be required for the determination of the temperature of any of the petroleum products for which it is to be used. The chamber must have quick acting closures at both top and bottom, so that they can be operated by a jerk of the cord or non-sparking chain used to raise and lower the assembly in the tank. When the closures are open, there must be a free path for product in the tank to flow through the chamber and past the thermometer bulb. When closed, the chamber must remain full.

(d) **Armored Case.**—This assembly is illustrated in Fig. 3, the armored case being made of non-sparking metal of not more than 0.5 in. outside diameter.

(e) **Angle Stem.**—The angle stem thermometer is installed in thermometer wells in the tank, as illustrated in Fig. 4. The glass stem of the thermometer shall be at least 3 ft. long and protected with a light metal tube. The sensitive portion of the thermometer shall not exceed 2.5 in. and the stem may have an angle of 90 deg. or greater to conform with the contour of the tank shell. The assembly is attached to the well by a threaded

---

[2] 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

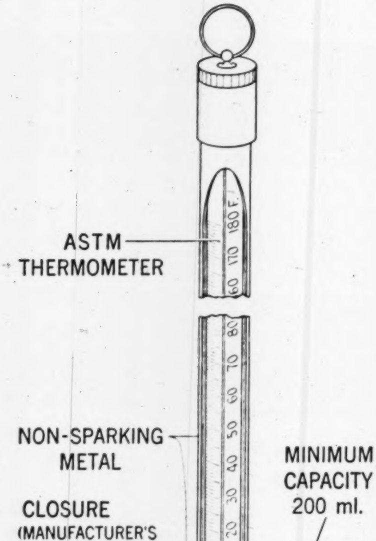

ASTM THERMOMETER
160 170 180 F.
0 10 20 30 40 50 60 70 80
NON-SPARKING METAL
MINIMUM CAPACITY 200 ml.
CLOSURE (MANUFACTURER'S DESIGN VARIES)
3" MIN.
1" ±1/8"
2" MIN.

FIG. 2.—TYPICAL FLUSHING CASE ASSEMBLY.

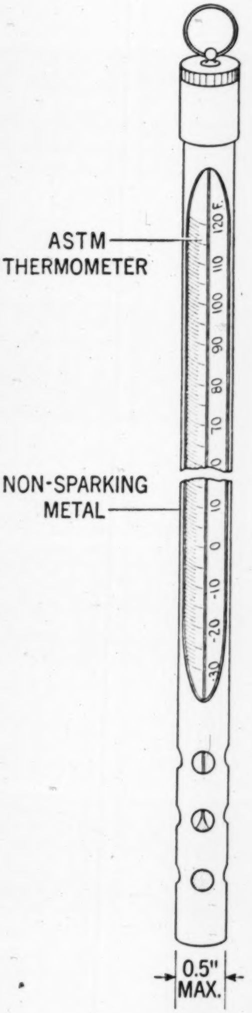

ASTM THERMOMETER
120 F.
110
100
90
80
70
0
NON-SPARKING METAL
10
0
-10
-20
-30
0.5" MAX.

FIG. 3.—TYPICAL ARMORED CASE ASSEMBLY.

coupling. It is permissible to use a thermometer with a separate graduated metal scale, provided the markings on the metal scale are permanently engraved, and provided temperature lines at 80 F. intervals are etched on the glass stem of the thermometer and match the corresponding lines on the metal scale.

Thermometer Standardization

5. (a) **Laboratory Inspection.**—Before initial use and at least once a year thereafter each thermometer should be standardized in the laboratory by comparing it with a standard thermometer at three or more temperatures, to make sure that it is accurate within the limits given in Table I.

(b) **Field Inspection.**—Before using a thermometer, check it in the field at one or more temperatures near mid-scale against a recently standardized thermometer. Make this check by placing the two thermometers side by side, leaving them undisturbed for at least ½ hr. before making comparative readings. As thermometers are precision instruments handle them carefully and examine them frequently. Avoid the use of a thermometer which has lost a substantial amount of black pigment from its engraved scale, because of the increased difficulty in obtaining readings. Never use a thermometer with a broken mercury column. If the mercury has been completely rejoined the thermometer may be used, provided it is found to be accurate within the limits of Table I by checking it against a standardized thermometer.

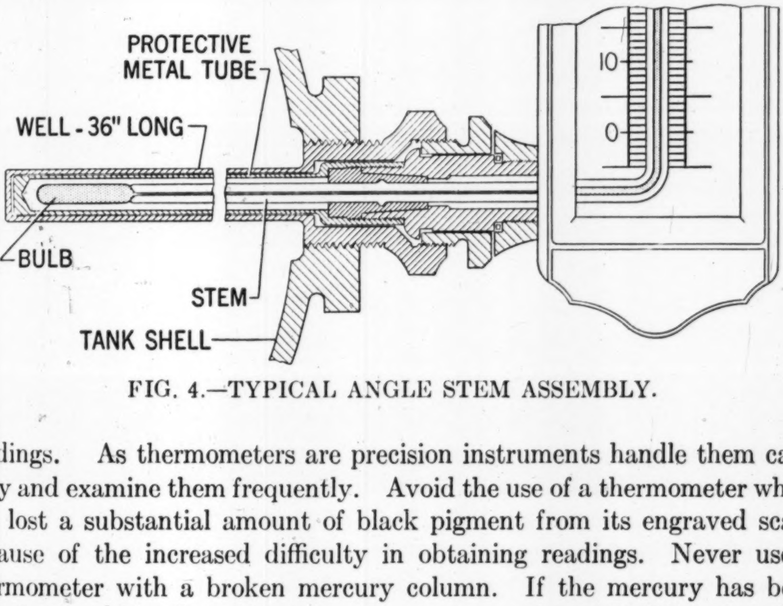


FIG. 4.—TYPICAL ANGLE STEM ASSEMBLY.

TANK EQUIPMENT

Non-Pressure Tanks

6. For products in tanks of the non-pressure type, temperature measurements are normally obtained by lowering a thermometer assembly through a roof hatch. The hatch through which temperature measurements are

to be taken should be located far enough from the edge of the tank to avoid local heating or cooling effects. It is suggested that this point be kept in mind when new tanks are built.

Pressure Tanks

7. (a) Thermometer Wells.—For products in tanks of the pressure type, temperatures cannot be obtained through a hatch. Therefore, such measurements are normally taken with thermometers in wells suitably located. Pressure tank cars and trucks should be equipped with thermometer wells which extend through the shell of the tank into the product. For ship and barge tanks, at least four compartments should be equipped with individual thermometer wells, each well to extend from a point 6 in. above deck through the deck to at least the center of the compartment. Horizontal cylindrical tanks should be equipped either with a vertical thermometer well through the center of the tank to about 6 in. from the bottom, or with two horizontal wells, one in the middle of the tank, the other 1 ft. above the bottom. Tanks of the spherical or spheroidal type may be equipped with either thermometer wells or pressure gaging locks. If horizontal thermometer wells are used, three are sufficient for a tank less than 30-ft. high. For a tank over 30 ft. high, at least four thermometer wells are necessary, one about 2 ft. above the lower capacity mark, one about 4 ft. below the upper capacity mark, and the other two at about equal intervals between the top and bottom wells.

(b) Pressure Lock.—A pressure sampling and gaging lock is a vapor-tight assembly mounted on the top of the tank. The lock must have a vapor-tight window and enclose a bob and tape on a reel which can be operated by a handle. A shut-off valve is required between the lock and the tank to prevent escape of vapors when the lock is open to the atmosphere.

PROCEDURE

Temperature Determinations

8. (a) As the average temperature of a product is required to calculate its volume at a standard temperature, it is important to determine temperatures accurately. Be sure that thermometers have attained the temperature of the product before reading them. Avoid change in the thermometer indication which may easily be caused by atmospheric temperature. Read thermometers to the nearest degree Fahrenheit. The temperature of a liquid product in storage may vary throughout its depth and from the center to the edge of the tank. If two or more temperatures are necessary at different levels, or if two or more series of such readings are made, average the readings arithmetically, round off the result to the nearest whole degree, and report it as the average temperature for the entire volume of the product.

(b) **Selection of Thermometer Assembly.**—Select a thermometer, Table I, of suitable range for the temperature to be measured and use the appropriate assembly as specified in Table II for various types of tanks and carriers. Follow the procedure described in the appropriate Paragraphs (*c*) to (*f*).

(c) **Cup Case.**—To obtain a temperature reading with the cup case assembly lower it through the hatch or pressure lock to the required level, Table III, and allow it to remain at this level at least as long as the minimum time specified in Table IV. The cup must be kept full when withdrawing the assembly to read the thermometer. If the atmospheric temperature differs by more than 20 F. from that of the liquid in the tank, empty the cup and again lower the assembly to the required level. To obtain a reading, raise the assembly and read the thermometer quickly with the cup sheltered below the edge of the hatch to avoid change of reading which may be caused by wind or atmospheric temperature. Record the temperature immediately. After use in heavy lubricating, fuel, or residual oils, clean all parts of the assembly with naphtha, kerosine, or gas oil and dry them with a cloth to avoid the formation of an insulating film of heavy oil.

(d) **Flushing Case.**—When using the flushing case assembly, open the top and bottom closures of the chamber and flush it two or three times in the upper portion of the product in the tank. Then lower the assembly to the required level, Table III. Keeping the chamber open at top and bottom, raise and lower the assembly through a distance of about 2 ft. for at least 2 min. Close the top and bottom openings. Raise the assembly, and read the thermometer quickly with the chamber sheltered below the edge of the hatch. Record the temperature immediately. After use in heavy lubricating, fuel, or residual oils, clean the thermometer and case with naphtha, kerosine, or gas oil and dry them with a cloth to avoid the formation of an insulating film of heavy oil.

(c) **Armored Case.**—The armored case assembly is used in vertical thermometer wells. The wells must be kept filled with a liquid such as kerosine or mineral seal oil of low volatility and freezing point. For horizontal cylindrical pressure tanks, tank cars and trucks, and pressure tanks on ships and barges, which are equipped with vertical thermometer wells, lower the thermometer assembly into the well to the middle of the product in the tank. For large tanks equipped with vertical wells, lower the thermometer to the desired level, Table III. Always let the thermometer remain at the required level for at least 5 min. Withdraw the assembly, but keep the perforated end in the well to avoid change of the thermometer indication. Read the thermometer quickly and record the temperature immediately.

TABLE II.—THERMOMETER ASSEMBLIES AND TEMPERATURE LEVELS FOR TANKS AND CARRIERS.

Tanks	Temperature Measurement Facilities	Thermometer Assembly	Temperature Levels
Storage:			
Fixed Roof	Roof Hatch	Cup or Flushing	See Table III
Floating Roof	Gaging Hatch	Cup or Flushing	See Table III
Variable Vapor Space	Gaging Hatch	Cup or Flushing	See Table III
	Thermometer Wells	Angle Stem	All wells 6 in. below surface of product
Pressure	Thermometer Wells:		
	Vertical	Armored	See Table III
	Horizontal	Angle Stem	All wells 6 in. below surface of product
	Pressure Lock	Cup or Flushing	See Table III
Horizontal Cylindrical:			
Non-Pressure	Gaging Hatch	Cup or Flushing	Middle of product
Pressure	Thermometer Wells:		
	Vertical	Armored	Middle of product
	Horizontal	Angle Stem	Middle of product and 1 ft. above bottom
Ship and Barge:			
Non-Pressure			
Not Heated	Deck Hatch	Cup or Flushing	Middle of product
Heated	Deck Hatch	Cup or Flushing	See Table III
Pressure	Thermometer Wells	Armored	Middle of product
Cars and Trucks:			
Non-Pressure	Dome Hatch	Cup or Flushing	Middle of product
Pressure	Thermometer Well	Armored	Middle of product

(f) **Angle Stem.**—Angle stem thermometers are permanent installations in thermometer wells except when removed for replacement or standardization, Section 5 (*a*).

TABLE III.—MINIMUM NUMBER OF TEMPERATURE MEASUREMENTS FOR VARIOUS DEPTHS OF PRODUCT.

DEPTH OF PRODUCT	MINIMUM NUMBER OF TEMPERATURE MEASUREMENTS	MEASUREMENT LEVELS
More than 15 ft.	3	3 ft. below top surface, middle, and 3 ft. above bottom surface of product.
10 to 15 ft.	2	3 ft. below top surface, and 3 ft. above bottom surface of product.
Less than 10 ft.	1	middle of product.

TABLE IV.—MINIMUM IMMERSION TIME FOR CUP CASE ASSEMBLY.

PRODUCT	MINIMUM IMMERSION TIME, MIN.
Gasolines and naphthas	5
Kerosines and gas oils of Saybolt Universal Viscosity less than 100 sec. at 100 F.	5
Light lubricating and fuel oils of Saybolt Universal Viscosity above 100 sec. at 100 F. and below 170 sec. at 210 F.	15
Heavy lubricating and residual oils of Saybolt Universal Viscosity between 170 and 500 sec. at 210 F.	30
Heavy cylinder, gear, and residual oils of Saybolt Universal Viscosity above 500 sec. at 210 F.	30
Greases	30

SPECIAL INSTRUCTIONS

Non-Pressure Tanks

9. If a tank is equipped with steam coils to heat the product, be sure that the steam has been turned off at least 1 hr. before obtaining temperature measurements for volume correction. For tank cars so equipped, make such measurements for liquid products before applying the steam. If the product is solid at ambient temperature, heat it until completely liquid, turn off the steam, and let the temperature equalize for 2 hr. before making temperature measurements. If a vertical cylindrical tank has both a center hatch and one near the shell, it is recommended that temperatures be obtained through both hatches. When obtaining measurements through a hatch near the shell, suspend the thermometer assembly as far from the shell as possible. If a ship or barge cargo consists of a single product, obtain temperatures in at least three compartments. If a cargo consists of more than one product, obtain temperatures for each product as follows:

Number of Compartments Containing One Product	Minimum Number for Temperature Measurements
1 or 2	each
3 to 6	2
7 or more	3

Pressure Tanks

10. Use a cup or flushing case assembly when obtaining temperatures through a pressure lock. Wind the tape on the reel until the bob is above the shut-off valve, lock the reel, *and close the valve to prevent escape of vapors from the tank.* Open the window, remove the bob, and attach the thermometer assembly to the tape. Close the window and open the valve. Follow the procedure of either Section 8 (*c*) or (*d*), lowering the thermometer assembly to the prescribed level with the reel. To make a reading, withdraw the assembly from the tank into the pressure lock and read the thermometer through the window immediately. After all required temperatures have been obtained, *be sure to close the valve before opening the window* to remove the thermometer assembly and replace the bob.

APPENDIX XI

PROPOSED METHOD OF ANALYSIS OF CALCIUM AND BARIUM PETROLEUM SULFONATES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of analysis covers procedures for the determination of mineral oil, calcium and barium sulfonate, calcium and barium carboxylate, calcium and barium hydroxide, combining weight, and specific gravity of crude and refined calcium and barium petroleum sulfonates by means of double decomposition with sodium carbonate.

Outline of Method

2. This method of analysis is outlined in Fig. 1.

Definition

3. The combining weight of calcium or barium sulfonate is the weight of calcium or barium sulfonate per gram-atom of calcium or barium, respectively.

Apparatus

4. (*a*) *Vacuum Oven* capable of operating at 70 to 100 C. under 3 mm. of mercury absolute pressure.

(*b*) *Beaker*, glass, with inverted rim, turned in 10 mm. from wall of beaker and pointed downward to a point 5 mm. below shoulder, 65 to 75 mm. in diameter, 75 to 85 mm. in height, and wall thickness (approximately 1 mm.) such that weight is less than 70 g.

(*c*) *Steam Bath.*

(*d*) *Muffle Furnace* capable of operating at 800 to 1000 C.

(*e*) *Atmospheric Ovens* capable of being maintained at 120 C. and at 70 to 80 C.

(*f*) *Mixing Cylinder*, 250-ml. capacity.

(*g*) *Water Bath* capable of being maintained at 40 to 50 C. and at 25 ± 0.2 C.

(*h*) *Vacuum Desiccator*, shielded.

(*i*) *Distillation Apparatus* as described in A.S.T.M. Method D 95, Test for Water in Petroleum Products and Other Bituminous Materials.[2]

(*j*) *Dish*, platinum, 100-ml. capacity.

(*k*) *Pycnometer* as shown in Fig. 3 of A.S.T.M. Method D 855, Analysis of Petroleum Sulfonates.[2] To calibrate, weigh to 1 mg. with cap in place, then fill with distilled water at 15 to 20 C. and place in a water bath at 25 ± 0.2 C. After 30 min., adjust the water meniscus at the top of the neck so it is exactly level. (To obtain a flat meniscus add a minute amount of wetting agent to the water surface.) Remove the pycnometer from the bath, and dry the outside. Cap and weigh to 1 mg. Record the water weight as W_c.

(*l*) *Weighing Bottle.*

Reagents

5. (*a*) ***Sodium Carbonate, c.p.***, anhydrous.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1948.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

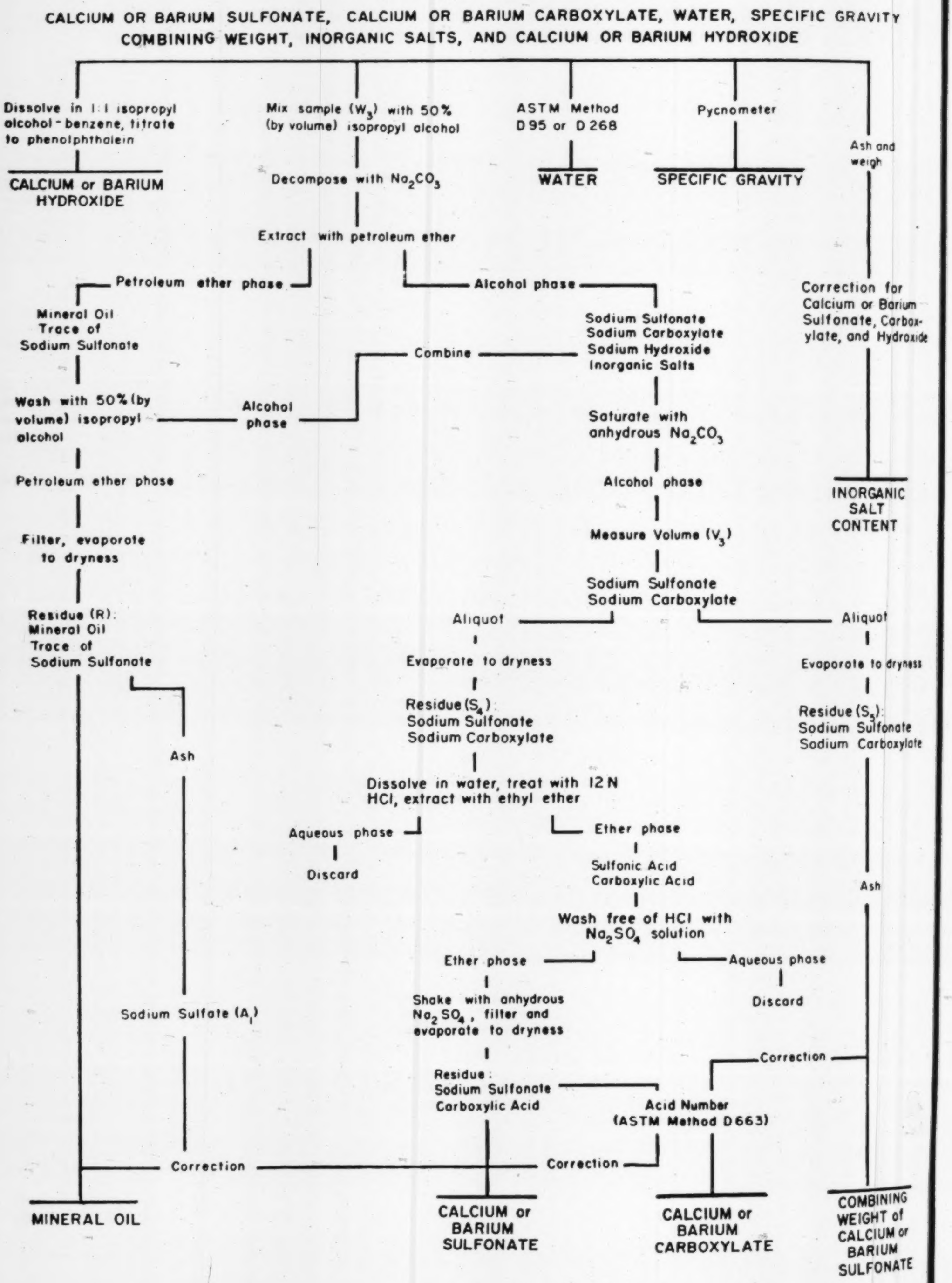

CALCIUM OR BARIUM SULFONATE, CALCIUM OR BARIUM CARBOXYLATE, WATER, SPECIFIC GRAVITY
COMBINING WEIGHT, INORGANIC SALTS, AND CALCIUM OR BARIUM HYDROXIDE
Dissolve in 1:1 isopropyl alcohol-benzene, titrate to phenolphthalein
CALCIUM or BARIUM HYDROXIDE
Mix sample (W_3) with 50% (by volume) isopropyl alcohol
Decompose with Na_2CO_3
Extract with petroleum ether
ASTM Method D95 or D268
WATER
Pycnometer
SPECIFIC GRAVITY
Ash and weigh
Petroleum ether phase
Alcohol phase
Mineral Oil
Trace of Sodium Sulfonate
Sodium Sulfonate
Sodium Carboxylate
Sodium Hydroxide
Inorganic Salts
Combine
Correction for Calcium or Barium Sulfonate, Carboxylate, and Hydroxide
Wash with 50% (by volume) isopropyl alcohol
Alcohol phase
Saturate with anhydrous Na_2CO_3
Petroleum ether phase
Alcohol phase
INORGANIC SALT CONTENT
Filter, evaporate to dryness
Measure Volume (V_3)
Sodium Sulfonate
Sodium Carboxylate
Residue (R):
Mineral Oil
Trace of Sodium Sulfonate
Aliquot
Aliquot
Evaporate to dryness
Evaporate to dryness
Residue (S_4)
Sodium Sulfonate
Sodium Carboxylate
Residue (S_3):
Sodium Sulfonate
Sodium Carboxylate
Ash
Dissolve in water, treat with 12 N HCl, extract with ethyl ether
Aqueous phase
Discard
Ether phase
Sulfonic Acid
Carboxylic Acid
Ash
Wash free of HCl with Na_2SO_4 solution
Ether phase
Aqueous phase
Discard
Sodium Sulfate (A_1)
Shake with anhydrous Na_2SO_4, filter and evaporate to dryness
Correction
Residue:
Sodium Sulfonate
Carboxylic Acid
Acid Number (ASTM Method D663)
Correction
Correction
MINERAL OIL
CALCIUM or BARIUM SULFONATE
CALCIUM or BARIUM CARBOXYLATE
COMBINING WEIGHT of CALCIUM or BARIUM SULFONATE

FIG. 1.—Outline of Method of Analysis of Calcium and Barium Petroleum Sulfonates.

(*b*) *Sodium Sulfate, c.p.*, anhydrous, crystalline.

(*c*) *Sodium Sulfate Solution, c.p. (20 per cent).*

(*d*) *Hydrochloric Acid, c.p. (12 N, and 0.5 N standard).*

(*e*) *Sulfuric Acid, c.p. (36 N).*

(*f*) *Methyl Orange Indicator Solution (0.1 per cent, aqueous).*

(*g*) *Phenolphthalein Indicator Solution (1 per cent in 50 per cent ethyl alcohol).*

(*h*) *Acetone, c.p.*

(*i*) *Benzene, c.p*

(*j*) *Ethyl Alcohol, U.S.P. (95 per cent).*

(*k*) *Ethyl Ether, U.S.P.*

(*l*) *Isopropyl Alcohol, refined grade (99 and 50 per cent by volume).*

(*m*) *Petroleum Ether.*—Boiling range, 30 to 75 C.; maximum nonvolatile impurities, 0.002 per cent.

Conversion of Calcium or Barium Sulfonate to Sodium Sulfonate

6. Introduce 125 ml. of isopropyl alcohol (50 per cent) into a 250-ml. separatory funnel. Add by means of a weighing bottle approximately 10 g. of sample (W_3) weighed to the nearest 0.01 g. and dissolve this in the alcohol. Warm the funnel and contents to 40 to 50 C. under a hot water tap or by immersion in a hot water bath. Add about twice the calculated amount of anhydrous sodium carbonate necessary for the conversion. Shake the contents of the funnel vigorously and then allow to cool.

Mineral Oil

7. (*a*) Extract the alcoholic solution from Section 6 six times with 30-ml. portions of petroleum ether, using two 250-ml. separatory funnels and collecting the petroleum ether extracts in a 500-ml. separatory funnel. Retain the interface "cuff," which includes the insoluble calcium carbonate, with the ether phase. When analyzing samples of barium sulfonate, retain the interface "cuff" with the alcohol phase. Drain the oil-free alcoholic sulfonate solution into a 250-ml. mixing cylinder. Rinse the 250-ml. separatory funnels with two 10-ml. portions of isopropyl alcohol (50 per cent) and add to the mixing cylinder. Wash the combined ether extracts with 50 ml. of isopropyl alcohol (50 per cent); add the wash and the calcium carbonate intermediate layer to the mixing cylinder.

(*b*) Filter the combined ether extracts through a small plug of cotton, placed in the vortex of a filter funnel, into a tared, inverted-rim beaker. Wash the separatory funnel and filter with 20 to 30 ml. of fresh petroleum ether, adding the washings to the inverted-rim beaker. Evaporate the ether solution to dryness on a steam bath, heating for 15 min. after the disappearance of the odor of petroleum ether or alcohol. Cool to room temperature and bring to constant weight in a vacuum desiccator at room temperature and under 3 mm. or less of mercury pressure, applying vacuum gradually and venting frequently to avoid spattering at the start. Denote the residue as *R*.

NOTE.—If it is known that a sample contains oil that is volatile under conditions of the test, it may be preferred to subtract the total of other constituents from 100 per cent to obtain the mineral oil content.

(*c*) Weigh a 1 to 2-g. sample (*r*) of the oil residue from Paragraph (*b*) into a tared, ignited platinum dish. Heat with a bunsen burner until the contents ignite and burn; then maintain at such a temperature that only ash and carbon remain after burning ceases. Ignite over a burner, or in a muffle furnace at 800 to 1000 C., until the oxidation of carbon is complete. Cool and add 3 to 4 drops of 36 *N* H_2SO_4. Fume off the H_2SO_4 over a burner in a hood, but do not heat strongly enough to cause spitting.

When fuming ceases, heat in a muffle furnace at 800 to 1000 C. to constant weight (A_1).

Sulfonate

8. (*a*) Warm the mixing cylinder (Section 7(*a*)) in a water bath at 40 to 50 C. Weigh out 18 g. of anhydrous sodium carbonate per 100 ml. of alcohol solution in the cylinder. Carefully admit a few grains to the cylinder, allowing the dissolved petroleum ether to evaporate slowly. When danger of boiling is past, add the remainder of the carbonate and shake the mixture vigorously. Allow to stand for a few minutes and repeat the shaking. If there is no excess of the solid carbonate, add an additional 1 to 2 g. and repeat the vigorous shaking. Replace the mixing cylinder in the water at 40 to 50 C. until separation into two layers is complete. Swirl to remove solid particles from the upper phase.

(*b*) Cool the funnel to room temperature (3 to 4 hr.). Read the volume (V_3) of the upper (alcohol) layer.

(*c*) Pipet a 50-ml. aliquot of the clear alcohol layer into a tared 150-ml. beaker. Evaporate to dryness on a steam bath, adding small portions of isopropyl alcohol (99 per cent) (or acetone) to aid in the removal of water. Dry to constant weight (S_4) in a vacuum oven at 80 C. under less than 3 mm. of mercury pressure, or in an atmospheric oven at 120 C. Denote the residue as S_4; save for sodium carboxylate determination.

Carboxylate

9. (*a*) Dissolve the residue (S_4) from Section 8 (*c*) in two 100-ml. portions of water and transfer the solution quantitatively to a 500-ml. separatory funnel; wash out the beaker with two 25-ml. portions of water and add the washings to the separatory funnel. Add 50 ml. of 12 *N* HCl to the funnel and shake vigorously. Allow the solution to cool and then extract the liberated acids with two 100-ml. portions of ethyl ether. Combine the ether extracts in a 500-ml. separatory funnel. Wash the combined ether extracts free of HCl by shaking with two or more 50-ml. portions of sodium sulfate (20 per cent) containing methyl orange indicator. Discard the salt washings.

(*b*) Drain off as much water as possible from the washed ether extract. Lay the separatory funnel on its side and introduce about 10 g. of anhydrous sodium sulfate; make sure that the funnel mouth is free of sodium sulfate crystals before inserting the stopper. Shake the mixture vigorously for 3 or 4 min., venting frequently. Filter the ether solution, using a small plug of cotton in the vortex of a filter funnel, into a tared 250-ml. Erlenmeyer flask. Rinse the funnel and filter with 20 ml. of ethyl ether; add the rinsings to the main ether solution and evaporate free of ether on a steam bath.

(*c*) Determine the acid number on the residue from Paragraph (*b*) as follows: Add 4 or 5 drops of phenolphthalein indicator and 50 ml. of isopropyl alcohol (50 per cent), previously neutralized to phenolphthalein. Warm if necessary to dissolve the sample and titrate with standard 0.1 *N* sodium hydroxide solution to a definite pink color. If the sample is so dark in color that it cannot be satisfactorily tested in this manner, determine the acid number according to A.S.T.M. Method D 664, Test for Neutralization Value (Acid and Base Numbers) by Electrometric Titration.[2] Denote the acid number on the residue as *B*.

Calcium or Barium Hydroxide

10. Weigh approximately 10 g. of sample into a 250-ml. Erlenmeyer flask. Add 50 ml. of ethyl alcohol and 50 ml. of benzene, and swirl until dissolved. Add 4 or 5 drops of phenolphthalein indicator

solution and titrate with standard 0.5 N HCl to the end point.

Water

11. Determine the water content of the sample by A.S.T.M. Method D 95 or by A.S.T.M. Method D 268 – 48, Sampling and Testing Lacquer Solvents and Diluents.[3] If Method D 268 – 48 is used, a correction may be necessary for the amount of water produced by calcium and barium hydroxide during the Fischer titration. One mole of water is liberated per equivalent of alkali.

Average Combining Weight of Sulfonate

12. (*a*) Pipet 25 ml. of the oil-free, salt-free alcoholic sulfonate solution (Section 8 (*b*)) into a tared, ignited, platinum dish. Evaporate to dryness on a steam bath, adding small portions of isopropyl alcohol (or acetone) to aid in removal of water. Dry to constant weight (S_5) in a vacuum oven at 80 C. under less than 3 mm. of mercury pressure, or in an atmospheric oven at 120 C.

(*b*) Carefully heat the dish over a small flame until the contents ignite; maintain the temperature so that the contents will burn gently. Ignite over a burner or in a muffle furnace at 800 to 1000 C., cool, and add 3 or 4 drops of 36 N H_2SO_4. Heat carefully over a small flame until fuming ceases, taking care not to heat strongly enough to allow spitting. Cool, and add 3 or 4 drops of 36 N H_2SO_4. Fume off the acid, taking care to avoid spitting. When fuming ceases, heat in muffle a furnace at 800 to 1000 C. to constant weight (A_2).

Specific Gravity

13. Fill the pycnometer with the sample, warming to 70 to 80 C. in a closed vessel if necessary. Hold the pycnometer at 70 to 80 C. until all air bubbles have risen to the top. Place in a bath at 25 ± 0.2 C. for 2 hr., adding more sample if necessary so the pycnometer remains full. Remove excess product flush with the top of the pycnometer by means of a spatula. Remove the pycnometer from the bath, wipe the sides of the neck with a cloth moistened with alcohol, wipe dry, and adjust the cap into place. Thoroughly dry the outside of the pycnometer and weigh to the nearest 1 mg. Record the sample weight as W_s.

Calculations

14. (*a*) *Mineral Oil Content.*—Calculate the mineral oil content of the sample as follows:

Mineral oil content, per cent =

$$\frac{100R}{W_3} - \frac{200\,A_1 R C_2}{142.1 r\,W_3}$$

where:

W_3 = weight of sample in grams (Section 6),

R = weight of oil residue in grams (Section 7 (*b*)),

r = weight of oil residue taken for ashing in grams (Section 7 (*c*)),

A_1 = weight of sodium sulfate ash from oil residue in grams (Section 7(*c*)), and

C_2 = combining weight of sodium sulfonate (Paragraph (*e*)).

(*b*) *Calcium or Barium Sulfonate Content.*—Calculate the calcium or barium sulfonate content as follows:

Calcium or barium sulfonate content, per cent =

$$\frac{S_4 V_3 C_1}{W_3 C_2} + \frac{100\,A_1 R C_1}{142.1 r\,W_3} - D$$

where:

S_4 = weight of sulfonate and sodium carboxylate in grams (Section 8 (*c*)),

V_3 = volume of dehydrated alcohol layer in milliliters (Section 8 (*b*)),

---

[3] 1948 Supplement to Book of A.S.T.M. Standards, Part II.

W_3 = weight of sample in grams (Section 6),
R = weight of oil residue in grams (Section 7 (*b*)),
r = weight of oil residue taken for ashing in grams (Section 7 (*c*)),
A_1 = weight of sodium sulfate ash from oil residue in grams (Section 7 (*c*)),
D = percentage of calcium or barium carboxylate content of sample (Paragraph (*c*)),
C_1 = combining weight of calcium or barium sulfonate (Paragraph (*e*)), and
C_2 = combining weight of sodium sulfonate (Paragraph (*e*)).

(*c*) *Calcium or Barium Carboxylate Content.*—Calculate the acid number of the residue (Section 9 (*c*)) as follows:

$$\text{Acid number, } B\text{, mg. of KOH per g.} = \frac{56HN_1}{S_4}$$

where:

H = volume of sodium hydroxide consumed in milliliters (Section 9 (*c*)),
N_1 = normality of sodium hydroxide, and
S_4 = weight in grams of residue (Section 8 (*c*)).

Calculate the carboxylate content as follows:

$$\text{Calcium carboxylate, } D\text{, per cent} = \frac{1.06\,B}{180}\,\frac{(S_4\,V_3\,C_1)}{(W_3\,C_2)}$$

$$\text{Barium carboxylate, } D\text{, per cent} = \frac{1.22\,B}{180}\,\frac{(S_4\,V_3\,C_1)}{(W_3\,C_2)}$$

where:

B = acid number of the residue (Section 9 (*c*),
S_4 = weight of sulfonate and carboxylate residue in grams (Section 8 (*c*)),
V_3 = volume of dehydrated alcohol layer in milliliters (Section 8 (*b*)),
W_3 = weight of sample in grams (Section 6),
C_1 = average combining weight of the sulfonate as the calcium or barium salts (Paragraph (*e*)), and
C_2 = average combining weight of the sulfonate as sodium salts (Paragraph (*e*)).

(*d*) *Calcium or Barium Hydroxide Content.*—Calculate the free alkalinity as calcium or barium hydroxide, as follows:

$$\text{Free alkalinity as calcium hydroxide, } M\text{, per cent} = \frac{3.7\,VN_2}{W_4}$$

$$\text{Free alkalinity as barium hydroxide, } M\text{, per cent} = \frac{6\,VN_2}{W_4}$$

where:

V = volume of hydrochloric acid consumed in milliliters (Section 10),
N_2 = normality of hydrochloric acid, and
W_4 = weight of sample in grams (Section 10).

(*e*) *Average Combining Weight of Sulfonate.*—Calculate the sodium carboxylate correction for the purified sulfonate as follows:

$$\text{Sodium sulfate equivalence of sodium carboxylate, } G\text{, per cent} = 0.127\,B$$

where:

B = acid number of the residue (Section 9 (*c*)),

Calculate the ash on the oil-free, salt-free soaps (Section 8 (*c*)) as follows:

$$\text{Ash, } F\text{, per cent} = \frac{100\,A_2}{S_5}$$

where:

A_2 = ash on oil-free, salt-free soap solution in grams (Section 12 (*b*)), and

S_5 = weight in grams of dry oil-free, salt-free soaps (Section 12 (*a*)) or one half the value S_4 (Section 8 (*c*)).

Calculate the combining weight of the sodium sulfonate as follows:

Combining weight of sodium sulfonate =

$$C_2 = \frac{7100}{F - G}$$

where:

F = percentage of ash on the oil-free, salt-free soaps, and

G = percentage of sodium sulfate equivalence of sodium carboxylate.

Calculate the combining weight of the calcium sulfonate as follows:

Combining weight of calcium sulfonate =

$$C_1 = 2\,C_2 - 6$$

Calculate the combining weight of the barium sulfonate as follows:

Combining weight of barium sulfonate =

$$C_1 = 2\,C_2 + 91$$

(*f*) *Specific Gravity.*—Calculate the specific gravity as follows:

$$\text{Specific gravity, 25/25 C.} = \frac{W_s}{W_c}$$

where:

W_s = weight in grams of sample contained by pycnometer at 25 C. (Section 13),

W_c = weight in grams of water contained by pycnometer at 25 C. (Section 4 (*k*)).

APPENDIX XII

PROPOSED METHOD OF TEST FOR BENZENE AND TOLUENE BY ULTRAVIOLET SPECTROPHOTOMETRY[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of benzene and toluene in petroleum products having a boiling range of 100 to 250 F. and a maximum content of 25 per cent total benzene plus toluene. Paraffins, naphthenes, or mono-olefins do not interfere with the analysis. Any interference that might arise from ultraviolet absorption due to diolefins or sulfur compounds is substantially eliminated by the base-line technique or by chemical treatment of the sample before analysis. The boiling range specification eliminates the xylenes, ethyl benzene, and styrene.

Definitions and Symbols

2. (*a*) *Transmittance*, τ is the ratio of the radiant power transmitted by a sample, P, to the radiant power incident on the sample, P_0, both being measured at the same spectral position and with the same slit width. In symbols:

$$\tau = \frac{P}{P_0}$$

Under the conditions of this method:

$$\text{Transmittance} = 10^{-abc}$$

where a, b, and c are defined in Paragraphs (*c*) to (*e*).

(*b*) *Absorbance*, A, is the logarithm to the base 10 of the reciprocal of the transmittance. A_1 and A_2 represent the absorbance of individual components.

Under the conditions of this method:

$$A = \log \frac{1}{\tau} = abc$$

(*c*) *Absorptivity*, a, is the ratio of the absorbance to the product of the concentration and the length of optical path. In symbols:

$$a = \frac{A}{cb}$$

Base-line absorptivity is the absorptivity for the sample measured from the base line vertically to the absorption peak.

(*d*) *Length of Optical Path*, b, is the distance in centimeters traversed by the beam of radiation in passing through the sample.

(*e*) *Concentration*, c, is the quantity of absorbing substance per unit volume. In this method it is expressed in grams per liter.

(*f*) *Calibration Coefficient*, K, is the ratio of the absorbance of a pure compound to its concentration. This quantity has significance only with respect to a particular absorption cell that is used both for calibration and the analysis of

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, October, 1948.

mixtures. Base-line calibration coefficient is the calibration coefficient for the pure compound measured from the base line vertically to the absorption peak.

(*g*) mμ is the symbol for millimicron.

(*h*) λ is the symbol for wave length.

Outline of Method

3. Benzene and toluene in petroleum products are determined by absorbance measurements on the sample at known dilution with a spectrophotometer that previously has been calibrated with solutions of pure benzene and toluene. Interference by conjugated diolefins and traces of sulfur compounds is minimized by use of the base-line method, or interference may be substantially eliminated by washing the sample with alkaline $KMnO_4$ solution before analysis.

Apparatus

4. (*a*) *Spectrophotometer* equipped for liquid samples with two vitreous silica absorption cells having optical path lengths of 1 cm. The instrument shall be capable of measuring absorbance with an accuracy of 1.0 per cent or better in the spectral region between 280 and 240 mμ with a nominal band width of 1 mμ or less. The spectral positions shall be reproducible to within 0.1 mμ.

NOTE 1.—Details of the method as written refer to the Beckman quartz spectrophotometer, Model DU,[3] equipped with ultraviolet accessories. If it is located in an area in which the temperature varies more than 5 C., the filter-slide entrance mirror mounting block must be modified for temperature control and measurements. Other ultraviolet spectrophotometers can be used with suitable modifications of the details of the operating procedures.

(*b*) *Pipets* calibrated with *iso*octane for volume delivery.

Reagents, Solvents, and Materials

5. (*a*) *Isooctane* for use as spectroscopic solvent.[4]

NOTE 2.—Technical *iso*octane is a satisfactory base stock for the preparation of spectroscopic solvent. Allow about 4 or 5 liters of this material to percolate through a column of activated silica gel 2 to 3 in. in diameter and 2 to 3 ft. in depth. Collect only the portion of the solvent that has a transmittance compared to distilled water greater than 90 per cent over the entire spectral range of 240 to 300 mμ. Store the solvent in scrupulously clean glass-stoppered bottles and always keep them covered.

In general, it will be best to use a fresh portion of silica gel for each batch of spectroscopic solvent, but the gel can be reactivated by pouring 500 ml. of acetone through the column, draining, drying by suction, and heating the gel in thin layers in an oven at 400 C. until it turns white. Keep the activated silica gel in closed containers until just before it is used.

(*b*) *Silica Gel (200-mesh).*

(*c*) *Alkaline $KMnO_4$ Solution.*—Dissolve 50 g. of $KMnO_4$ and 25 g. of solid KOH in distilled water and dilute to 1 liter.

(*d*) *Lens Paper.*

(*e*) *Solvents for Cleaning Cells.*—Residue-free acetone or ethyl alcohol.

Reference Standards

6. (*a*) *Benzene.*—National Bureau of Standards standard hydrocarbon sample No. 210.

(*b*) *Toluene.*—National Bureau of Standards standard hydrocarbon sample No. 211.

Preparation of Apparatus

7. (*a*) *Preliminary Adjustments.*—Check, and if necessary, readjust the wave-length scale according to the directions furnished by the manufacturer of the instrument. With the slit fully open, focus the hydrogen lamp to obtain maximum intensity. The wave-length scale adjustment and focusing of the

---

[3] Manufactured by Nationa Technica Labs., South Pasadena, Calif.

[4] Spectroscopic solvent *iso*octane is available from the Chemical Products Dept., Phillips Petroleum Co., Bartlesville, Okla.

hydrogen lamp must be made with extreme care, since improper focusing of the hydrogen lamp may cause the calibration of the instrument to be in error.

Because of a change of absorbance with temperature, a difference of 5 C. in the temperature of the cell block between the time of calibration and the time of analysis can cause an error of as much as 1 per cent in the total aromatic content of the sample. If temperature fluctuations reach this value, the filter slide entrance mirror mounting block modified for temperature control must be used. When a sample whose temperature differs appreciably from that of the cell block is to be analyzed, allow the sample to remain in the cell block long enough for temperature equilibrium to be reached before taking readings.

(*b*) *Selection of Slit Widths.*—Use a slit width in accordance with the energy output of the instrument but not exceeding 0.5 mm. Use the same slit widths in the base-line spectral positions as are used at the adjacent key positions. If the instrument will not balance using the chosen slit widths, the mirrors shall be cleaned and readjusted or other steps taken to obtain the necessary energy.

(*c*) *Cleaning Cells.*—Clean the silica absorption cells by rinsing them thoroughly three times with acetone or alcohol and drying with a stream of clean dry air. Wipe the outside of the transparent surfaces with clean lens tissue. Examine these surfaces and use the cells only if they are absolutely clean.

NOTE 3.—Acetone has absorption in the same spectral region as benzene and toluene, and therefore drying must be complete.

Calibration of Spectrophotometer

8. (*a*) *Preparation of Solutions.*—Proceeding as directed in Section 10 (*a*), dilute weighed samples of pure benzene and pure toluene to give the approximate concentrations of benzene and toluene given in the following table. Prepare these solutions within one day of the time they are to be used.

Approximate Spectral Position, mμ	Benzene, g. per l.	Toluene, g. per l.
275	30 to 60	5.0 to 11.0
268.5	4.0 to 8.0	0.20 to 0.45
267	5.0 to 12.0	0.34 to 0.80
259	1.0 to 2.0	0.20 to 0.45
254.5	0.17 to 0.37	0.22 to 0.50
252.5	0.65 to 1.5	0.25 to 0.55

(*b*) *Location of Key Positions and Base-Line Positions.*—Using suitable concentrations of both benzene and toluene, read the absorbance, compared to spectroscopic solvent in the solvent cell, at 2-mμ intervals in the range 245 to 275 mμ. In the neighborhood of the benzene minima near 252.5 and 259 mμ and the benzene maximum near 254.5 mμ, take readings with the benzene at 0.1-mμ intervals. Similarly, in the neighborhood of the toluene minimum near 267 mμ and the toluene maximum near 268.5 mμ, take readings with the toluene at 0.1-mμ intervals. Because these maxima and minima are quite sharp, it is necessary that the spectral positions be located carefully. Use the slit widths determined in Section 7(*b*). Select the above-determined minima points and 275.0 mμ as the base-line positions for the analysis and the above determined maxima points as the key positions for the analysis.

(*c*) *Measurement of Absorbance.*—Read the absorbance (as described in Section 10(*b*)), compared to spectroscopic solvent in the solvent cell, of the appropriate dilution prepared in accordance with Paragraph (*a*) at the key positions and corresponding base-line positions determined in accordance with Paragraph (*b*). The cells shall be kept covered.

(*d*) *Determination of Cell Correction.*—Read the absorbance, compared to spectroscopic solvent in the solvent cell, of spectroscopic solvent in the sample cell at each of the key positions and base-line positions. This absorbance is a measure of the difference in the two silica cells.

(*e*) *Calculation of Calibration Coefficients.*—From the known purity of the sample, the weight of the sample, and the dilution, calculate the actual concentration of benzene or toluene in grams per liter for each of the above solutions. For each measurement at the key positions and base-line positions, calculate the corrected absorbance by subtracting the cell correction obtained as described in Paragraph (*d*) from the corresponding observed absorbance (Paragraph (*c*)). Calculate the calibration coefficients for each position, this coefficient being the ratio of corrected absorbance to concentration of the sample in grams per liter. Recheck those data which differ from the average coefficient at each position by more than the percentage indicated in the following table:

Approximate Spectral Position, mμ	Permissible Deviations	
	From Average Benzene *K* values, per cent	From Average Toluene *K* values, per cent
275	10.0	1.5
268.5	1.5	0.5
267	1.5	1.0
259	1.0	0.5
254.5	0.5	0.75
252.5	1.0	0.75

Average calibration coefficients for all points except benzene at 275 mμ shall be based on absorbances in the range of 0.40 to 1.0. Absorbances for calibration shall be obtained at each spectral position from at least two different weighings and four different dilutions from each weighing for each pure compound. The dilutions may or may not be of the same concentration.

(*f*) *Calculation of Base-Line Calibration Coefficient.*—From the calibration coefficients, calculate the base-line calibration coefficients for the two key points for each compound as indicated below. Use for the calculations the average calibration coefficient for each compound at each spectral position.

$$(K_B)_1 = K_{B_{254.5}} - \left[K_{B_{259}} + \frac{259 - 254.5}{259 - 252.5} (K_{B_{252.5}} - K_{B_{259}}) \right]$$

$$(K_B)_2 = K_{B_{268.5}} - \left[K_{B_{275}} + \frac{275 - 268.5}{275 - 267} (K_{B_{267}} - K_{B_{275}}) \right]$$

$$(K_T)_1 = K_{T_{254.5}} - \left[K_{T_{259}} + \frac{259 - 254.5}{259 - 252.5} (K_{T_{252.5}} - K_{T_{259}}) \right]$$

$$(K_T)_2 = K_{T_{268.5}} - \left[K_{T_{275}} + \frac{275 - 268.5}{275 - 267} (K_{T_{267}} - K_{T_{275}}) \right]$$

where:

$(K_B)_1$, $(K_B)_2$ = base-line calibration coefficients of benzene at base line 1 (benzene peak) and base line 2 (toluene peak), respectively,

$(K_T)_1$, $(K_T)_2$ = base-line calibration coefficients of toluene at base line 1 (benzene peak) and base line 2 (toluene peak), respectively,

$K_{B_{254.5}}$, $K_{B_{268.5}}$, etc. = calibration coefficients of benzene at spectral positions indicated by the numbers, and

$K_{T_{267}}$, $K_{T_{275}}$, etc. = calibration coefficients of toluene at the spectral positions indicated by the numbers.

These data will be valid only for the instrument on which they have been obtained and only as long as the optical system stays in adjustment. The absorption maxima should be checked every three months, or more often if there is any reason to suspect a displacement of the wave-length scale.

Preparation of Sample

9. Provided the final boiling point of the material exceeds 250 F., charge 500 ml. of the material to a still and distill the sample at atmospheric pressure in such manner as to meet the following requirements:

(*a*) Discard any distillate boiling below 100 F. Collect, for ultraviolet analysis, the distillate boiling in the range 100 to 250 F. Collect for testing the quality of the fractionation, 1 per cent portions (5-ml. cuts) of distillate starting at the 250 F. vapor-temperature point until the minimum absorptivity point is reached.

(*b*) Test the 5-ml. cuts for absorptivity by ultraviolet spectrophotometry as follows: Blend the first cut, if necessary, so as to read an absorbance not greater than 1.0, following the procedure described in Section 10 (*a*). Read the absorbance at 261.3 mμ of the sample in the sample cell, compared to spectroscopic solvent in the solvent cell, using the same slit width as is used at about 267 mμ. Calculate the absorptivity of this test portion as the ratio of the corrected absorbance to the concentration of the blend in grams per liter.

(*c*) If the absorptivity exceeds 0.3, put 10 ml. of the blended cut in a glass-stoppered mixing cylinder with 20 ml. of alkaline $KMnO_4$ and shake on a mechanical shaker for 20 min. Fill the sample cell by means of a pipet with a portion of the hydrocarbon layer. Do not wash the blend with water after washing with $KMnO_4$. Read the absorbance at 261.3 mμ of the sample in the sample cell compared to alkaline $KMnO_4$-washed spectroscopic solvent in the solvent cell, using the same slit width as is used at about 267 mμ. Calculate the absorptivity and if it exceeds 0.3, test the other cuts in the same manner.

(*d*) If an absorptivity of 0.3 or less is not obtained, the distillation shall be repeated on a more efficient column or at higher reflux ratio. If a value of 0.3 or less is obtained on one of the cuts, combine in aliquot proportions the unblended portions of it and the lower boiling cuts, if any, together with the 100 to 250 F. cut, so that a single sample containing no C_8 aromatics can be analyzed.

NOTE 4.—Fenske-packed column 6 ft. in height, operated at a 10:1 reflux ratio, usually will make the required separation. Extreme care shall be used in the handling of samples for spectroscopic analysis. Minute quantities of many substances will seriously affect the results. Contact with cork or rubber stoppers or with the ordinary stopcock lubricants shall be avoided.

NOTE 5.—If the original material contains more than 50 per cent aromatics as determined by the proposed Method of Test for Olefinic and Aromatic Hydrocarbons in Gasoline,[5] add to the charge to the distillation 50 ml. of nonaromatic hydrocarbon boiling between 240 and 260 F. to act as a "spacer" between toluene and the C_8 aromatics.

Procedure

10. (*a*) *Dilution of Sample.*—An occasional sample may not require blending, in which case pipet a portion of the sample into the sample cell and measure the absorbance at the two key positions. If the absorbance is 1.0 or less at both positions, proceed with the analysis as

---

[5] See Appendix IX to the Report of Committee D-2, *Proceedings*, Am.Soc. Testing Mats., Vol. 47, p. 333 (1947).

described in Paragraph (*b*). If the absorbance is greater than 1.0 at either point, add 10 to 15 ml. of spectroscopic solvent to a clean, dry, glass-stoppered 25- to 50-ml. volumetric flask. Weigh out approximately 1 g. of sample, or more if necessary, in the flask and dilute to volume with spectroscopic solvent. Accurately dilute this blend, if necessary, with spectroscopic solvent, using 2-, 3-, 4-, 5-, or 10-ml. pipets and 25- or 50-ml. glass-stoppered volumetric flasks until the total concentration of benzene and toluene is such that the absorbance at all spectral positions falls in the range of 0.4 to 1.0. More accurate data will result if the concentration of the sample is adjusted to give approximately the same absorbance as that obtained in the benzene calibration This may require two or more different blends If the approximate concentration of the sample is known, time can be saved by calculating the proper dilution.

NOTE 6.—In order to avoid contamination, extreme care shall be used in the dilution and the handling of diluted samples for spectroscopic analysis. Weathering of sample and blends shall be avoided.

(*b*) *Measurement of Absorbance*—Pipet portions of the blend into the sample cell of the spectrophotometer. Cover the cells immediately to prevent transfer of aromatic hydrocarbons from the sample cell to the solvent cell. Check the windows of the absorption cells and make certain they are clean. Measure the absorbance by rapidly repeating in order the dark-current setting, the sensitivity setting, and the absorbance reading until it is apparent that drifts in the instrument are not causing incorrect readings. Record the absorbance of the sample as compared to the solvent at the spectral positions selected in Section 8(*b*), using the same slit widths as used in the calibration, and at 232 mμ, using the maximum slit width. Calculate the absorptivity at 232 mμ; if this value exceeds 0.25, the sample shall be washed with alkaline $KMnO_4$ according to the alternate procedure described in Section 11. The concentrations of all blends shall be such that the absorbances as read on the instrument at all points will be in the range 0.4 to 1.0 (except at 275 mμ).

(*c*) *Determination of Cell Correction.*—Measure and record the absorbance of the solvent-filled sample cell as compared to the solvent-filled solvent cell.

Alternate Procedure

11. (*a*) *Treatment with Alkaline $KMnO_4$ Solution.*—Blend the sample, if blending is necessary, as directed in Section 10(*a*). Place 10 ml. of the sample dilution used for analysis in a 50-ml. glass-stoppered mixing cylinder, add 20 ml. of the alkaline $KMnO_4$, and shake in a mechanical shaker for 20 min.; avoid longer shaking. This treatment removes unsaturates and sulfur compounds. In the same manner, treat a portion of the same spectroscopic solvent used in the dilution with some of the same alkaline $KMnO_4$.

(*b*) *Measurement of Absorbance.*—Follow the procedure described in Section 10(*b*) and (*c*).

Calculation

12. (*a*) *Absorptivities.*—Calculate the absorptivities at each of the spectral positions employed as follows:

$$a = \frac{A_c}{cb}$$

where:

a = absorptivity,

A_c = corrected absorbance (observed absorbance minus cell correction),

c = concentration of sample in grams per liter, and

b = length of optical path (cell thickness) in centimeters (in this case $b = 1$).

(*b*) *Base-Line Absorptivities.*—Calculate the base-line absorptivity at each of the two key positions as described in Section 8(*f*).

(*c*) *Concentrations of Benzene and Toluene.*—Using the principle that, on a standard concentration basis, the absorbances of benzene and toluene are additive, calculate the concentration of benzene and toluene in the sample by solution of the following linear simultaneous equations written for the absorption at the two base-line points involved.

$$a_1 = \left(\frac{C_B}{100}\right)(K_B)_1 + \left(\frac{C_T}{100}\right)(K_T)_1$$

$$a_2 = \left(\frac{C_B}{100}\right)(K_B)_2 + \left(\frac{C_T}{100}\right)(K_T)_2$$

These equations may be solved simultaneously for C_B and C_T as follows:

$$C_T = \left[\frac{100(K_B)_2}{(K_T)_1(K_B)_2 - (K_T)_2(K_B)_1}\right] a_1 - \left[\frac{100(K_B)_1}{(K_T)_1(K_B)_2 - (K_T)_2(K_B)_1}\right] a_2$$

$$C_B = \left[\frac{100(K_T)_2}{(K_T)_2(K_B)_1 - (K_T)_1(K_B)_2}\right] a_1 - \left[\frac{100(K_T)_1}{(K_T)_2(K_B)_1 - (K_T)_1(K_B)_2}\right] a_2$$

where:

a_1, a_2 = base-line absorptivities of the mixture for base line 1 (benzene peak) and base line 2 (toluene peak), respectively,

$(K_B)_1$, $(K_B)_2$ = base-line calibration coefficients of benzene for base line 1 (benzene peak) and base line 2 (toluene peak), respectively,

$(K_T)_1$, $(K_T)_2$ = base-line calibration coefficients of toluene for base line 1 (benzene peak) and base line 2 (toluene peak), respectively, and

C_B, C_T = concentration in percentage by weight of benzene and toluene, respectively.

Solve these equations directly for percentage of benzene and toluene on the portion of the original sample used for spectroscopic analysis. Calculate the concentrations of benzene and toluene as percentages by weight on the original sample basis, as follows:

$$\text{Benzene, per cent} = C_B \times \frac{W_1}{W_2}$$

$$\text{Toluene, per cent} = C_T \times \frac{W_1}{W_2}$$

where:

W_1 = weight of that portion of sample used for spectroscopic analysis (usually the 100 to 250 F. portion), and

W_2 = weight of original charge to the distillation flask.

REPORT OF COMMITTEE D-3[*]
ON
GASEOUS FUELS

Two meetings of Committee D-3 on Gaseous Fuels and its seven subcommittees, as well as two meetings of its Advisory Committee, were held during the past year. Subcommittees were most active as shown by seven meetings of these groups held during this period.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 – 46 T) be approved for reference to letter ballot of the Society for adoption as standard with revisions, as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Collection of Gaseous Samples (E. O. Mattocks, chairman) has prepared a revised draft of a proposed tentative method for sampling liquefied petroleum gases, embodying suggestions received on the first draft. Following full subcommittee approval and in conjunction with Committee D-2 on Petroleum Products and Lubricants, it is expected to have this method ready for consideration as a new tentative in the near future. Preparation of tentative methods for sampling natural gases and manufactured gases is under way. The method covering natural gases has already been discussed by the subcommittee and agreement reached on revisions to be included.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman) has distributed a third draft of the proposed method for measuring gaseous fuel samples to the members of the subcommittee and others interested for their review and comment.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. S. Jessup, chairman) prepared the proposed revisions of the Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 – 46 T), as appended to this report.[1] These revisions cover the following: (*1*) Changes in the specifications for calorimeter thermometers and flue thermometers, (*2*) addition of a new section giving directions for reducing results of calorific value determinations to the basis of measurements of gas in the field, which may be made under other than "standard" conditions, and (*3*) the omission of the partial derivation of the humidity correction formula as shown in the present method and insertion of reference to the complete derivation as given in the National Bureau of Standards *Circular C464*.

The subcommittee also approved of the adoption of this method as standard with the incorporation of the recommended revisions.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] This revised method was adopted as standard by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—The chairman of this group has endeavored during the past year to utilize the results obtained by the National Bureau of Standards in its exhaustive tests on specific gravity apparatus in preparing in acceptable form a tentative method for determination of specific gravity of gaseous fuels. It is hoped that this method will be completed and ready for committee approval during the present year.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (E. H. Smoker, chairman) has directed its activities for the past year almost entirely to the development of a standard method for determination of total sulfur in gaseous fuels. One method devised by the Institute of Gas Technology is now under consideration. Copies have been distributed to members of the group and comments requested.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—A proposed tentative method of moisture determination by the dew-point method previously submitted to members of the subcommittee by the chairman was extensively discussed at its last meeting. Practical experiences with the use of this method were reviewed and attention directed to complications which might be produced by the presence of certain constituents in the gas. While this method was not felt suitable for all gases, no other was currently available to which some limitations did not apply. In considering the applicability of this method it was generally agreed that moisture determinations were of much less importance with manufactured than with natural gases. Following redrafting of the method to include necessary changes, it is proposed to circulate it to members for comment.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman). The following two reports prepared by this subcommittee were published during the year: "Cooperative Analysis of a Standard Sample of Natural Gas with the Mass Spectrometer,"[3] by Martin Shepherd, and "Analysis of a Natural Gas by Volumetric Chemical Methods and by the Mass Spectrometer."[4]

At the two meetings held by this subcommittee, decision was reached to prepare for study tentative methods for analysis of carbureted water gas and of natural gas by chemical-volumetric methods and a mass spectrometric method for analysis of natural gas. An initial draft of the latter method was discussed by the subcommittee at its March meeting and necessary revisions approved for inclusion in a second draft.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. W. Gauger.
Vice-Chairman, E. F. Schmidt.
Secretary, K. R. Knapp.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

K. R. KNAPP,
Secretary.

---

[3] *Journal of Research*, Nat. Bureau Standards, Vol. 38, No. 5, May, 1947, p. 491 (*RP*1789).
[4] *Analytical Chemistry*, Vol. 19, p. 635 (1947).

REPORT OF COMMITTEE D-4*

ON

ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials has held three meetings during the past year: in Atlantic City, N. J., on June 18, 1947; in Chicago, Ill., on November 6, 1947; and in Washington, D. C., on March 4, 1948.

Subsequent to the 1947 Annual Meeting, Committee D-4 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Tentative Specifications for:

Asphalt Cements for Use in Pavement Construction (D 946 – 47 T).

Hot-Mixed, Hot-Laid Asphaltic Concrete Base and Surface Courses (D 947 – 47 T).

Tentative Revision of Standard Method of Test for:

Distillation of Cut-Back Asphaltic Products (D 402 – 36).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new tentatives together with the tentative revision of D 402 appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

I. NEW TENTATIVES

Specifications for Asphaltic Mixtures for Sheet Asphalt Pavements.[1]—Committee D-4 recommends that these specifications developed by Subcommittee C-10 on Plant-Mix Bituminous Surfaces and Bases (Fred Hubbard, chairman) be accepted for publication as tentative. These specifications represent a continuation of the preparation of specifications for bituminous paving mixtures initiated last year by the development of the Tentative Specifications for Hot-Mixed, Hot-Laid Asphaltic Concrete Base and Surface Courses (D 947 – 47 T).

Specifications for Emulsified Asphalt.[1]—The committee recommends that these specifications be accepted for publication as tentative. These specifications were prepared by Subcommittee C-4 on Specifications for Emulsified Asphalts (F. H. Baumann, chairman) as a consolidation of five existing standard specifications without modification of technical requirements; it is anticipated that, when adopted as standard, the recommended specifications will replace the five existing Standard Specifications D 397, D 398, D 399, D 401, and D 631.

Methods for Sampling Bituminous Paving Mixtures.[1]—The committee recommends that these methods be accepted for publication as tentative. These methods were developed by Subcommittee B-15 on Sampling Road Materials (C. E. Proudley, chairman) to provide a suitable procedure to which reference may be made in specifications for bituminous paving mixtures under the jurisdiction of Committee D-4.

II. REVISION OF TENTATIVES

Tentative Methods of Sampling Bituminous Materials **(D 140 – 46 T)**.[2]—Committee D-4 joins with Committee D-8 on Bituminous Waterproofing and Roofing Materials in recommending the following revision in these methods. This revision was approved by Committee D-8 in June, 1947.

New Section.—Add the following as a

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] These specifications and methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

[2] 1946 Book of A.S.T.M. Standards, Part II.

new Section 1 on Scope and renumber the subsequent sections accordingly: "1. *Scope.*—These methods apply to the sampling of liquid, semisolid or solid bituminous materials at the point of manufacture, and from bulk storage, tank cars, distributors, barrels, drums, or cakes."

Tentative Specifications for Hot-Mixed, Hot-Laid Asphaltic Concrete Base and Surface Courses (**D 947 - 47 T**).[3]—The committee recommends the following revisions in these specifications:

Title.—Add the word "for" between the words "concrete base."

Table II.—Interchange the series of grading requirements under the headings "Grading No. 1" and "Grading No. 2."

Section 4—Add the following sentence and note at the end of this section:

"The penetration grades shall be 60–70, 70–85, 85–100, 100–120, or 120–150.

NOTE.—The exact penetration grade to be used depends on the type of construction, climatic conditions, and amount and nature of traffic, and the required grade should be specified."

Section 7(d).—Add the following at the end of the second sentence: "in which case the clearance shall be so adjusted as to prevent undue breakage of the coarse aggregate during the mixing operation."

Delete the words "or box" in the last sentence of this paragraph.

Section 9 (b).—Insert the following sentence at the end of this paragraph: "The temperature between these limits shall be regulated according to the penetration grade of the asphalt used."

Section 9 (c).—Change the third sentence to read as follows, by the addition of the italicized words: "The temperature between these limits shall be regulated according to the *penetration grade of the asphalt*, temperature of the atmosphere and the workability of the mixture."

Table III.—Change the heading of bitumen requirements to read as follows by the addition of the italicized words: "Bitumen, per cent *by Weight* of Total Mixture."

III. TENTATIVE REVISIONS OF STANDARDS

Committee D-4 recommends the following tentative revisions in two standards:

Standard Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (**D 544 - 41**):[2]

Section 4 (e).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*e*) Boiling in Hydrochloric Acid.—In the case of cork and self-expanding cork (types I and II) expansion joint fillers only, the test specimen [when boiled for 1 hr. in hydrochloric acid (sp. gr. 1.19)] shall show no evidence of disintegration *when immersed in hydrochloric acid (sp. gr. 1.19) and boiled for 1 hr.* Discoloration, or a small amount of swelling shall not be considered as failure. *The expansion joint fillers (types I and II) shall be examined immediately after the end of the boiling period and shall be rejected for any of the following evidences of disintegration: (1) particles of cork dislodged during the boiling period, (2) specimen lacks resiliency, is very friable, and easily broken to pieces, or (3) surface of specimen appears porous and particles of cork are easily dislodged by rubbing with fingers.*

Section 7 (c).—Add the following sentence to the end of this paragraph: "Tests shall be made on expansion joint fillers that have not been deformed or broken in handling."

Standard Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (**D 545 - 41**):[2]

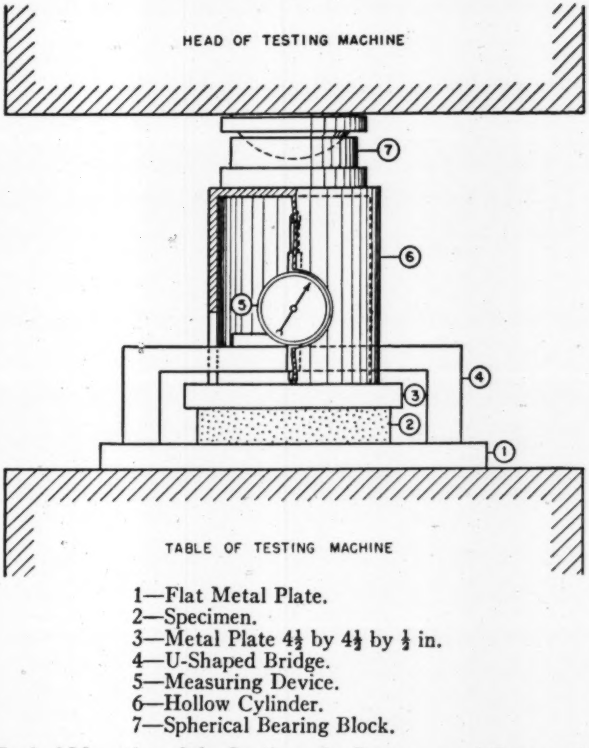
Section 3.—Add the following sentence to the end of this section: "The test specimens shall be prepared for further testing as described in Section 2 (*b*)"

---

[3] 1947 Supplement to Book of A.S.T.M. Standards, Part II.

Section 4 (b).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) Mounting.—The test specimen shall be placed on a flat metal plate, and a 4½ by 4½ by ½-in. metal plate ground to have plane parallel faces shall be centered on the top surface of the specimen. A simple U-shaped bridge shall be employed to support a dial *gage* or other suitable measuring device reading to 0.001 in. above the center of the specimen. A *hollow* metal *load transfer* cylinder [or other device for transferring the load from the moving head of the testing machine around the measuring apparatus to the plate covering the specimen shall be placed upon the plate] *with slots for inserting the U-shaped bridge and an opening for reading the measuring device shall be placed between the moving head of the testing machine and the plate covering the specimen. A typical mounting is shown in Fig. 1 but other suitable devices may be used.* A spherical bearing block shall be mounted between the upper end of the cylinder and the moving head of the testing machine. *Both the hollow metal cylinder or other device and the spherical bearing block shall be accurately centered so that the load will be applied uniformly to the test specimen.*



1—Flat Metal Plate.
2—Specimen.
3—Metal Plate 4½ by 4½ by ½ in.
4—U-Shaped Bridge.
5—Measuring Device.
6—Hollow Cylinder.
7—Spherical Bearing Block.

FIG. 1.—Typical Mounting of the Specimen for Recovery and Compression Tests.

New Figure.—Add the accompanying Fig. 1 showing typical mounting of the specimen for recovery and compression tests.

Section 4.—Add a new Paragraph (*f*) to read as follows:

(*f*) *Loss of Bitumen.* In the case of bituminous fiber (type V) expansion joint fillers, the waxed paper, together with any bitumen squeezed out of the specimen during the compression test, shall be removed from the test specimen. The test specimen shall be accurately weighed to 0.1 g. and the loss of bitumen calculated.

Section 6.—Change the second sentence to read as follows by the addition of the italicized words: "The specimen shall be *immersed and* boiled for 1 hr. in hydrochloric acid (sp. gr. 1.19)."

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee D-4 concurs with the recommendation[4] made by Committee C-9 on Concrete and Concrete Aggregates that the Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 - 37) be revised by immediate adoption of a change in Section 2 (*a*).

IV. ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:[2]
Calcium Chloride (D 98 - 46 T),
Tentative Methods of:
Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 46 T),
Sampling and Testing Calcium Chloride (D 345 - 46 T), and
Test for Sulfonation Index of Road Tars (D 872 - 46 T).

The committee also recommends that the Tentative Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 47 T)[3] be approved for reference to letter ballot of the Society for adoption as standard with the following revisions. The purpose of these revisions is to regroup requirements for assembly of apparatus in a more logical sequence and to have requirements for reporting agree with those of the Standard Specifications for Cut-Back Asphalts, D 597 and D 598.

Section 1.—Change to read as follows by the addition of the italicized word and the omission of the one in brackets: "1. This method covers [the] *a* distillation test for the examination of cut-back asphaltic products."

Section 2.—Change the last sentence to read as follows by the addition of the italicized words and the omission of those in brackets: "The residue remaining after a temperature of 680 F. (360 C.) is reached [is reserved for additional tests] *may be tested by appropriate methods.*"

Section 3 (d).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*d*) *Shield.*—A galvanized iron shield lined with ⅛ in. asbestos *and* fitted with transparent covered windows, of the form and dimensions shown in Fig. 2 *is* used to protect the flask from air currents and to prevent radiation. The cover (top) *is* [may be of transite board] made in two parts *and may be of transite board* or [it may be] of galvanized iron lined with ⅛-in. asbestos.

Section 3 (e).—Insert a comma after "diameter" in the first sentence.

Section 6.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 6. (*a*) Support the flask, with the shield in place, on a tripod or ring over two sheets of 20-mesh wire gauze, 150 mm. square, as shown in Fig. 3. *Protect the burner from drafts by a suitable shield or chimney.* Connect the condenser tube by a tight cork joint. [Insert the thermometer through a cork in the neck, with the bottom of the bulb 6.5 mm. (¼ in.) from the bottom of the flask.] *The condenser tube shall be clean and dry.*

Section 6 (b).—Add the following sentence to the beginning of this paragraph: "Insert the thermometer through a cork in the neck, with the bottom of the bulb 6.5 mm. (¼ in.) from the bottom of the flask."

Section 6 (c).—Delete this paragraph.

Section 6 (d).—Reletter as Paragraph (*c*) and add the following sentence after the first sentence: "The distance from the neck of the flask to the outlet end of the adapter shall be not more than 700 nor less than 600 mm."

---

[4] See p. 264.

Retain the present second and third sentences of this paragraph as Paragraph (*d*).

Section 6 (*e*).—Delete this paragraph.

Section 7 (*a*).—Change to read as follows by the addition of the italicized words and the omission of the parenthesis in brackets:

> 7. (*a*) Weigh 200 ml., *the weight of which shall be* [( ] calculated from the specific gravity of the material at 60 F. (15.5 C.), of the sample into the 500-ml. flask. Assemble the apparatus and apply heat so *that* the first drop comes over in 5 to 15 min.

Section 8.—Change to read as follows by the addition of the italicized words and figures and the omission of the words in brackets:

> 8. The ***residue at 680 F. (360 C.), by difference from the total distillate,*** [results of the distillation test] shall be reported ***as*** [in] percentage by volume of ***the*** water-free material.
>
> The following fractions ***shall be*** [are] reported as percentages ***by volume*** of the total ***distillate*** [fraction] to 680 F. (360 C.):
>
> Up to 437 F. (225 C.),
> Up to 500 F. (260 C.), and
> Up to 600 F. (316 C.).
>
> Additional fractions may be specified, such as:
>
> Up to 320 F. (160 C.),
> Up to 347 F. (175 C.), and
> Up to 374 F. (190 C.).
>
> ***Results of tests on the distillation residue shall be reported as specified in the appropriate methods of the American Society for Testing Materials.***

V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Preformed Expansion Joint Fillers for Concrete (*Nonextruding and Resilient Types*) (**D 544 - 41**).[2]—The committee recommends, that the tentative revisions[4] of these specifications be approved for reference to letter ballot of the Society for adoption as standard. These tentative revisions, submitted in November, 1943, comprise revision of Section 1, addition of Paragraph (*d*) to Section 2, revisions of Section 4 (*a*), (*b*), and (*f*), and addition of Paragraphs (*g*) and (*h*) to this section.

Standard Methods of Testing Preformed Expansion Joint Fillers for Concrete (*Nonextruding and Resilient Types*) (**D 545 - 41**).[2]—The committee also recommends that the tentative revisions[5] of these methods be approved for reference to letter ballot of the Society for adoption as standard. These tentative revisions submitted in November, 1943, comprise revisions of Sections 1, 2 (*a*), 4 (*a*), 5 (*a*) and the addition of two new Sections 8 and 9. It is also recommended that Section 4 (*a*) be further revised by the addition of the following sentence to the end of the paragraph: "In the case of bituminous (type V) expansion joint fillers, the test specimen shall be weighed accurately to 0.1 g. and then placed between two sheets of waxed paper prior to being placed between the compression plates as described in Section 4 (*b*)."

VI. WITHDRAWAL OF STANDARD

In view of the recommendation for adoption as standard of the Tentative Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 47 T), Committee D-4 recommends that the Standard Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 36) be withdrawn.

VII. TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-4 and the joint subcommittee of Committees D-4 and D-18 have reviewed existing tentative specifications and methods of test which have been published by the Society for two years or more without revision, and Committee D-4 recommends that the following be continued as tentative, without revision:

---

[5] 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 455 and 456.

Tentative Specifications for:

Materials for Stabilized Base Course (D 556 - 40 T) and Materials for Stabilized Surface Course (D 557 - 40 T). Suggestions for changes in these two specifications have been made and are under consideration by the joint subcommittee of Committees D-4 and D-18.

Tentative Methods of Test for:

Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T). This method is in general use, but a subcommittee is actively engaged in a study of improved provisions and requirements.

Moisture-Density Relations of Soils (D 698 - 42 T). These tentative specifications are under the joint jurisdiction of Committee D-4 and Committee D-18 on Soils for Engineering Purposes. Subcommittees of the latter committee are considering revisions of this method which will be referred to Committee D-4 upon their approval.

The recommendations appearing in this report have been approved by letter ballot of Committee D-4, the results of which will be reported at the Annual Meeting.[6]

Committee D-4 is cooperating with Committee D-8 on Bituminous Waterproofing and Roofing Materials and with Society Headquarters in developing plans and a program for a symposium on the general subject of Durability Tests for Bituminous Materials, with a view to presentation at the 1949 Spring Group Meeting or the Annual Meeting of the Society.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, F. H. Baumann.
First Vice-Chairman, C. E. Proudley.
Second Vice-Chairman, A. T. Goldbeck.
Third Vice-Chairman, E. W. Klinger.
Secretary, B. A. Anderton.

This report has been submitted to letter ballot of the committee, which consists of 129 members; 99 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. J. EMMONS,
Chairman.

B. A. ANDERTON,
Secretary.

---

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Preformed Expansion Joint Filler for Concrete (Bituminous Type) (D 994 - 48 T), and Bituminous Paving Plant Requirements (D 995 - 48 T).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the new tentative specifications appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II.

REPORT OF COMMITTEE D-5*
ON
COAL AND COKE

Committee D-5 on Coal and Coke held meetings in Atlantic City, N. J., on June 20, 1947, and in Washington, D. C., on March 4, 1948. The Advisory Subcommittee held a meeting in Atlantic City, N. J., on June 20, 1947, and in Washington, D. C., on March 4, 1948.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. A. Selvig.
Vice-Chairman, D. H. Davis.
Secretary, C. H. Sawyer.

During the year three members were added to the committee and three members resigned, resulting in the present membership of 59, of whom 16 are classified as producers, 20 as consumers, and 23 as general interest members.

Subcommittee **XIX** on Designation of Coal Sizes (J. D. Doherty, chairman) was discharged as the subcommittee had completed its assignments by preparing the Standard Method of Test for Screen Analysis of Coal (D 410) and the Standard Method for Designating the Size of Coal from Its Screen Analysis (D 431).

I. NEW TENTATIVE METHOD

Committee D-5 recommends that the Tentative Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances, appended hereto,[1] be accepted for publication as tentative.

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

In view of the minor significance of the changes, described, Committee D-5 recommends that the following revisions of three standards be immediately incorporated in these standards. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (**D 271 – 46**):[2]

Figure 2 (*a*) *and* (*b*).—Delete the illustrations of the two riffle samplers and substitute the accompanying Fig. 1 (*a*) and (*b*).

Section 2 (*g*).—Change the requirements for size of divisions of the large riffle sampler from "$\frac{1}{2}$ or $\frac{5}{8}$-in." to read "$\frac{1}{2}$ or $\frac{3}{4}$-in."

Standard Methods of Sampling Coals Classed According to Ash Content (**D 492 – 46**):[2]

New Figures 1 and 2.—Add as new Figs. 1 and 2 the accompanying Figs. 2 and 3 showing types of riffle samplers suitable for the reduction procedure specified in Section 7 (*c*), (*d*), and (*e*).

Section 7 (*a*).—Omit the second sentence which reads as follows: "The riffle samplers specified in Paragraphs (*b*) to (*e*) shall be similar in type to those shown in Fig. 2 (*a*) and (*b*) of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials."

Section 7 (*c*).—Add the following sentence: "Two types of riffle samplers suitable for reducing the sample as specified are shown in Fig. 1."

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] This method was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

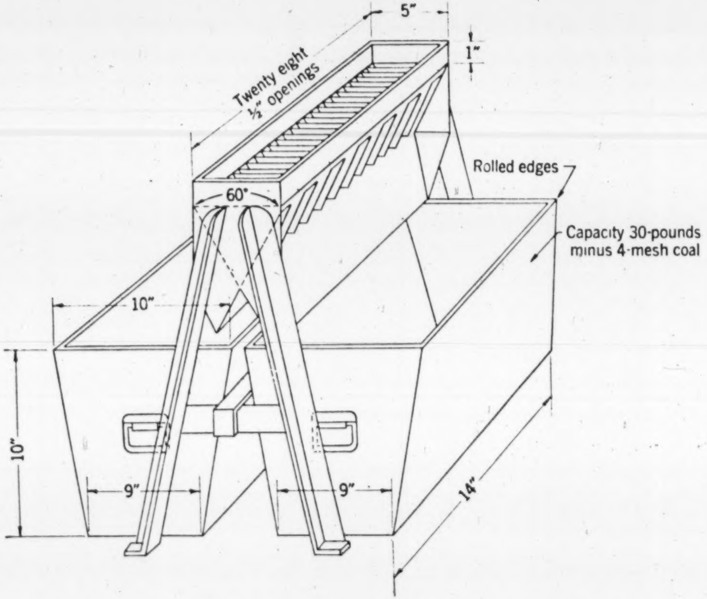
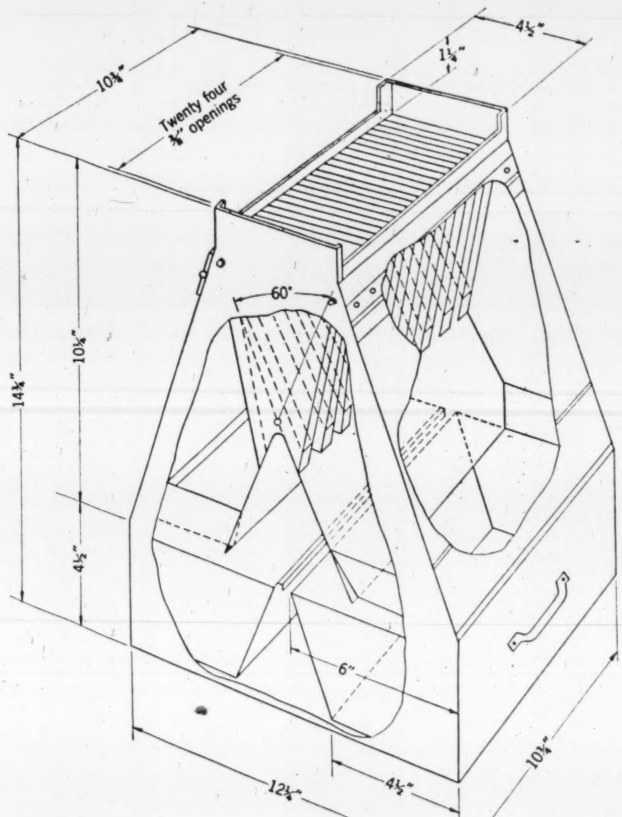
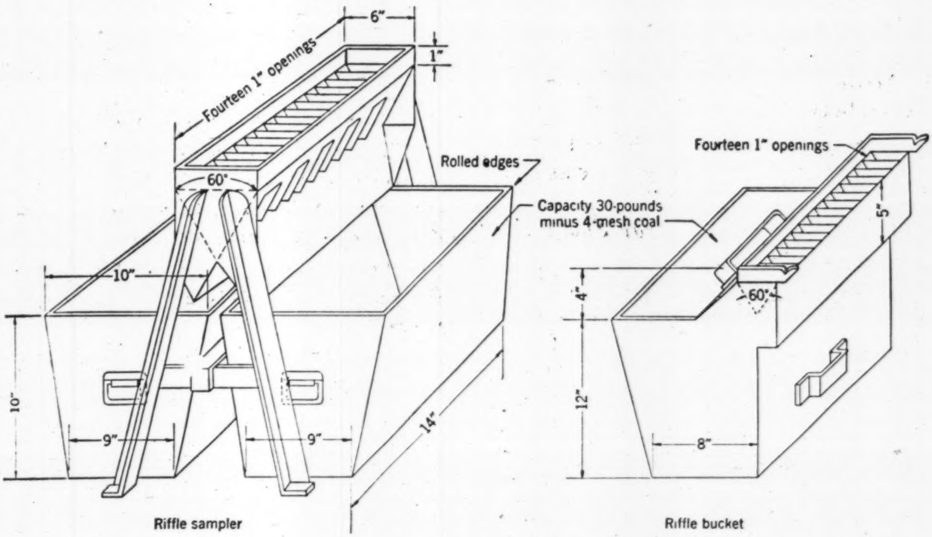

5"
1"
Twenty eight ½" openings
60°
Rolled edges
Capacity 30-pounds minus 4-mesh coal
10"
10"
9"
9"
14"

FIG. 1 (*a*).—Large Riffle Sampler.


4½"
1¼"
10¾"
Twenty four ⅜" openings
14¾"
10¼"
4½"
60°
6"
12¼"
4½"
10¼"

Note:
May be constructed as either closed
or open type. Closed type preferred.

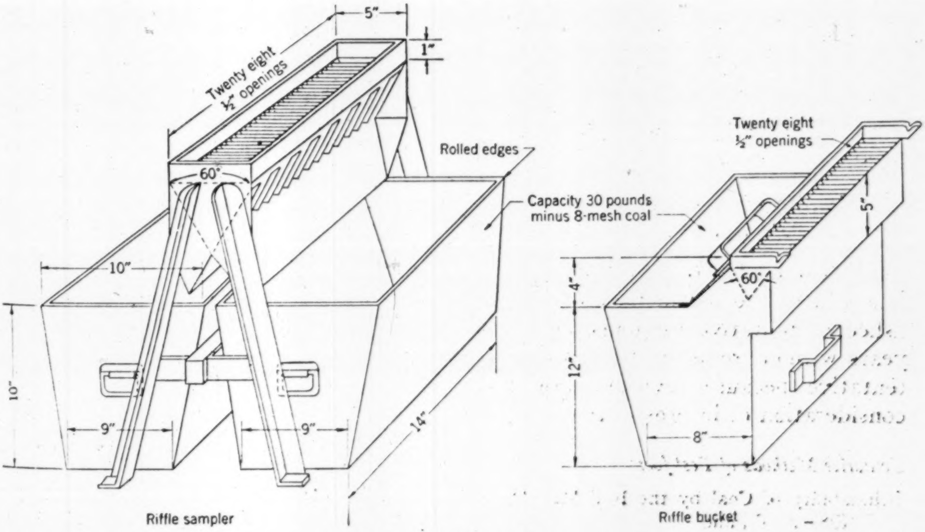
FIG. 1 (*b*).—Small Riffle Sampler.


6"
1"
Fourteen 1" openings
Rolled edges
60°
Capacity 30-pounds minus 4-mesh coal
10"
10"
9"
9"
14"
Fourteen 1" openings
5"
4"
60°
12"
8"

Riffle sampler

Riffle bucket

FIG. 2.—Types of Riffle Samplers Suitable for Reduction Procedure Specified in Section 7(*c*) of Method D 492.


5"
1"
Twenty eight ½" openings
Rolled edges
60°
Capacity 30 pounds minus 8-mesh coal
10"
10"
9"
9"
14"
Twenty eight ½" openings
5"
4"
60°
12"
8"

Riffle sampler

Riffle bucket

FIG. 3.—Types of Riffle Samplers Suitable for Reduction Procedure Specified in Section 7 (*d*) and (*e*) of Method D 492.

Section 7 (d).—Add the following sentence: "Two types of riffle samplers suitable for reducing the sample as specified are shown in Fig. 2."

Section 7 (e).—Add the following sentence between the third and fourth sentences: "Two types of riffle samplers suitable for reducing the sample as specified are shown in Fig. 2."

Standard Method of Drop Shatter Test for Coke (**D 141 – 23**):[2]

Figure 1.—Delete the present Fig. 1 showing the shatter test machine and substitute the accompanying Fig. 4. The new illustration shows a machine of improved design in that the vertical supports are mounted outside of the base plate making it easier to clean the machine after dropping the coke. Other improvements in construction make the machine more convenient to operate.

III. REVISION OF TENTATIVE

Tentative Method of Drop Shatter Test for Coal (**D 440 – 37 T**):[2]

The committee recommends that the accompanying Fig. 4 be substituted for the present Fig. 1 showing the shatter test machine for the reasons stated above under the revision of Standard Method D 141.

IV. TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following methods, which have been published as tentative for longer than two years without revision, be continued as tentative because revisions are under consideration or in prospect:

Tentative Methods of Test for:

Grindability of Coal by the Ball Mill Method (D 408 – 37 T), and

Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman) has created a Special Section on Revision of Ultimate Analysis Methods, under the chairmanship of O. W. Rees. This special section held a meeting in Cincinnati, Ohio, on October 21, 1947, and agreed to prepare revisions of the methods for determination of carbon, hydrogen, and nitrogen as given in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 46).

Subcommittee XIII on Coal Sampling (Henry F. Hebley, chairman) at a meeting held in Cincinnati, Ohio, on October 21, 1947, agreed to recommend as tentative the Method of Sampling Coal for Volatile Matter Determination in Connection with Smoke Ordinances.[1] The method is based on sampling experiments to determine the minimum gross sample required to give an accuracy for volatile matter of ± 1 per cent on the moisture- and ash-free basis. It is applicable to the sampling of relatively small quantities of coal as delivered to apartment buildings, hotels, laundries, domestic consumers, etc., where it is impracticable to collect large gross samples. The stipulation that volatile matter is to be reported on the moisture- and ash-free basis makes it practical to collect smaller gross samples than would be required for the same degree of accuracy if the analysis were reported on the customary as-received basis.

The subcommittee also recommended improvements in the construction of riffle samplers described earlier in this report. These improved designs should be readily adaptable for commercial manufacture.

---

[3] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee XVI on Ignitibility of Coal and Coke (R. A. Sherman, chairman).—Two papers covering research on laboratory methods of testing the reactivity of solid fuels to oxidizing gases were presented this year.[4] One of these papers covers research done by the Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.; the other, work done by the Combustion Research Section, Coal Branch, U. S. Bureau of Mines, Pittsburgh, Pa. Probably the application of such methods of ignition testing will continue to be along research lines until such time as reactivity indices as determined in the laboratory can be evaluated in terms of commercial uses of solid fuels.

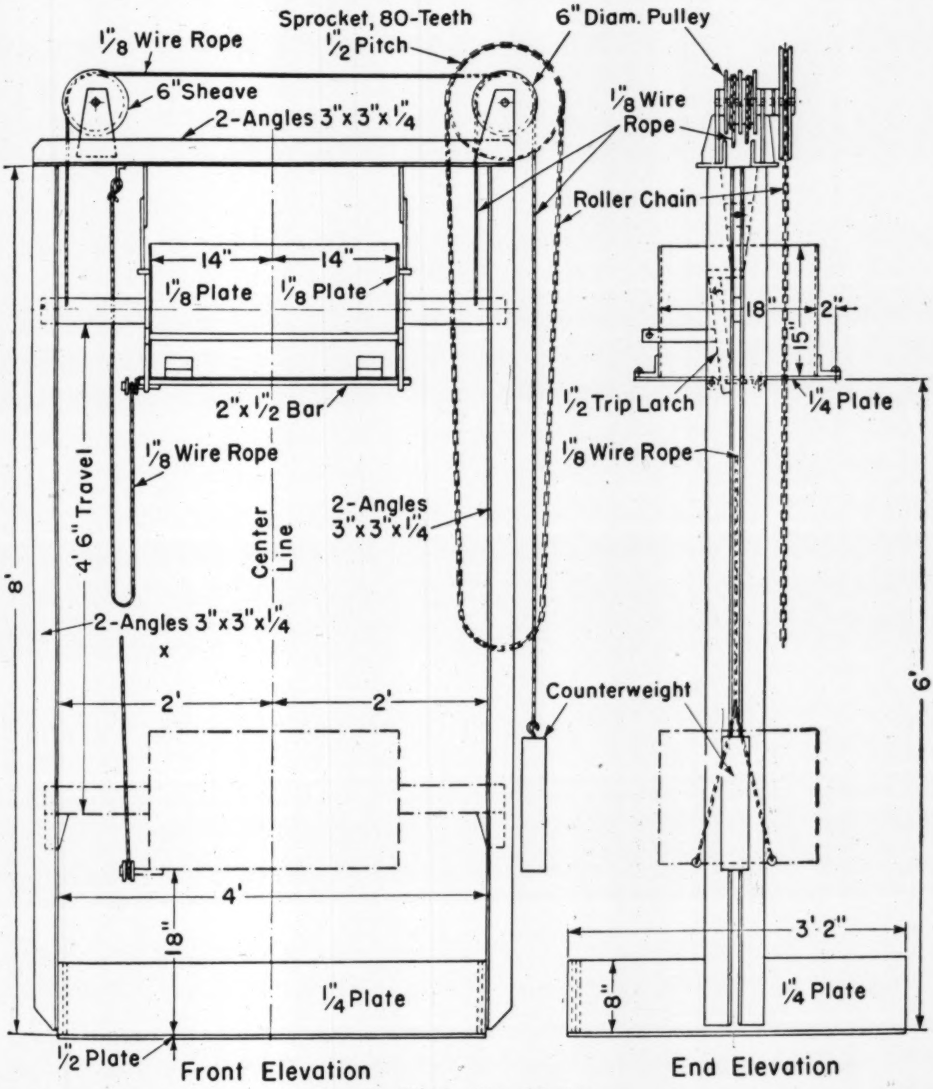

Sprocket, 80-Teeth
1/2" Pitch
6" Diam. Pulley
1/8" Wire Rope
6" Sheave
2-Angles 3"x3"x1/4
1/8" Wire Rope
Roller Chain
14"
14"
1/8" Plate
1/8" Plate
18"
2"
15"
2"x1/2" Bar
1/2" Trip Latch
1/4" Plate
1/8" Wire Rope
1/8" Wire Rope
4' 6" Travel
Center Line
2-Angles 3"x3"x1/4"
8'
2-Angles 3"x3"x1/4
x
6'
2'
2'
Counterweight
4'
18"
3' 2"
8"
1/4" Plate
1/4" Plate
1/2" Plate
Front Elevation
End Elevation

FIG. 4.—Shatter Test Machine.

---

[4] Orning, A. A., Mallov, S., and Neff, M., "Reactivity of Solid Fuels to Air and Oxygen," *Industrial and Engineering Chemistry*, Vol. 40, pp. 429–432 (1948).

Jonakin, J., Cohen, P., Corey, R. C., and Jain, B., "Measurement of the Reactivity of Solid Fuels by the Crossing Point Method," see p. 1269.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (J. B. Romer, chairman) held a meeting in Washington, D. C., on March 4, 1948. Some experimental work has been conducted on the problem of sampling pulverized coal from moving streams of coal and air inside pipes as the coal is enroute from the pulverizers to the furnaces. Further work is planned to obtain data regarding the efficiency and correctness of various types of sampling equipment and procedures. Work also has been planned for investigation of laboratory methods for determination of fineness by wet and dry sieving, resistance to air flow, hand sieving, and machine sieving.

This report has been submitted to letter ballot of the committee, which consists of 59 members; 50 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

REPORT OF COMMITTEE D-6[*]
ON
PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 19, 1947, at Atlantic City, N. J. and on February 27, 1948, at New York, N. Y.

The Advisory Committee held two meetings during the year: on November 7, 1947, at Albany, N. Y. and on February 27, 1948, at New York, N. Y. During the November meeting, a new Subcommittee V on Research was established.

Cooperative relations are being continued between Committee D-6 and other A.S.T.M. committees, as well as with other standardizing bodies.

At the present time, Committee D-6 consists of 80 members, of whom 64 are voting members; 25 are classified as producers, 25 as consumers, 24 as general interest, and 6 as consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. R. Willets
Vice-Chairman, M. A. Krimmel.
Secretary, H. A. Birdsall.

NEW TENTATIVE METHODS

The committee recommends that the following eight new methods, prepared by Subcommittee I, be accepted for publication as tentative, as appended hereto:[1]

Methods of Test for:

Reducible Sulfur in Paper,
Analytical Filter Papers,
Stretch of Paper and Paper Products Under Tension,
Organic Nitrogen in Paper and Paperboard,
Measuring Paraffin Wax Absorptiveness of Paper,
45 Degree, 0 Degree Directional Reflectance for Blue Light (Brightness) of Paper,
Water-Vapor Permeability of Paper, Paperboard, and Other Sheet Materials, which represents a consolidation and replacement of Tentative Methods D 783 and D 830.
Preparation of Magnesium-Oxide Standard for Spectral Reflectivity.

ADOPTION OF TENTATIVE METHODS AS STANDARD

The committee recommends that the following four tentative methods be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:[2]

Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 46 T),
Resistance of Paper to Passage of Air (D 726 – 46 T),
Tensile Breaking Strength of Paper and Paper Products (D 828 – 45 T), and
Wet Tensile Breaking Strength of Paper and Paper Products (D 829 – 45 T).

The committee also recommends that the Tentative Methods of Test for Ply Adhesion of Paper or Vulcanized Fibre (D 825 – 46 T)[2] be revised as follows and referred to letter ballot of the Society for adoption as standard:

Section 3. Letter the present paragraph as "(*a*)" and add the following as a new Paragraph (*b*):

(*b*) The second step in calibration is that of measuring the accuracy of indication when the jaw carriage is attached to the wheel as it is in testing paper. This is done by clamping a cord in the jaws of carriage, *C*, and passing it over a low-friction, ball-bearing pulley not less than 1.5 in. in diameter, and suspending calibration weights on the end of the cord to cover the range of the instrument. Assurance should be made that the friction of the pulley used is not great by passing the cord direct from the wheel which bears the scale horizontally to the pulley and calibrating this system against the original calibration as outlined in Paragraph (*a*). If a proper pulley has been chosen, the error due to the use of it should not be over 3 per cent of the weight applied. When assurance has been made that the pulley friction is no greater than 3 per cent, then the error due to carriage friction may be measured. If the carriage bearings are not worn or tight, the friction due to its horizontal movement will not be measurable. If the carriage bearing friction is appreciable, the instrument should be reconditioned.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] These methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

[2] 1946 Book of A..ST.M. Standards, Part III-B.

WITHDRAWAL OF TENTATIVE METHODS

The committee recommends that the following two tentative methods be withdrawn as they are being replaced by the new comprehensive Methods of Test for Water Vapor Permeability of Paper, Paperboard, and Other Sheet Materials:

Tentative Methods of Test for:

Water Vapor Permeability of Paper and Paperboard (D 783 – 44 T), and

Water Vapor Permeability of Paper and Other Sheet Materials at Elevated Temperature and Humidity (D 830 – 45 T).

EDITORIAL CHANGE IN STANDARD

The committee recommends that the Standard Method of Test for Thickness of Paper and Paper Products (D 645 – 43) be editorially revised by changing the scope of Method A as stated in Section 1 to read as follows by the addition of the italicized words: "Method A, for the general run of papers, paper products, and *for container grades of paperboard.*"

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (C. G. Weber, chairman) has held two meetings during the year. The subcommittee has been active in developing new methods of test and has also continued its critical study of various methods of test of paper and paper products that have been adopted by other agencies, for example, those of the Technical Association of the Pulp and Paper Industry. The eight new tentative methods and the four tentatives being recommended for adoption as standards are under the jurisdiction of this subcommittee.

Methods on the following subjects are being given consideration by the subcommittee:

Abrasion,
Fiber identification,
Gloss,
Linting,
Moisture expansivity,
Permanganate number,
Stiffness,
Total chloride content of paper, and
Zinc pigments in paper.

In addition to the above subjects, studies are being continued which may lead to proposed revisions in the Standard Methods of Test for Basis Weight of and Paper Products (D 646 – 44), and Hydrogen Ion Concentration (pH) of Paper Extract (D 778 – 46).

Subcommittee I also has developed a method for wax pick test for surface strength of paper. This is still under consideration and is, therefore, not being submitted for publication as tentative but is included in an Appendix to this report as information to elicit criticism.

Subcommittee III on Specifications for Paper (P. S. Wehmer, chairman) held two meetings during the year. This subcommittee is currently developing standard specifications for filter paper for chemical analysis.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee IV on Container Board (W. B. Lincoln, Jr., chairman) held two meetings during the year, and is currently working on the following test procedures:

Adhesion test of component parts of corrugated board,
Beam test,
Bursting strength of paperboard,
Flat crush test,
Immersion test and delamination of plies,
Ring crush test, and
Static bending test.

In addition to the above subjects, studies are being made which may lead to a proposed revision in the Tentative Method of Test for Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 - 44 T). Further study is also being made of the Proposed Method of Test for Scuff Resistance of Paperboard.[4]

This report has been submitted to letter ballot of the committee, which consists of 64 voting members; 44 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. REID,
Chairman.

G. H. HARNDEN,
Secretary.

---

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 356 (1947).

APPENDIX

PROPOSED METHOD OF WAX PICK TEST FOR SURFACE STRENGTH OF PAPER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is applicable to relatively smooth and dense coated and uncoated papers and is designed to measure the surface strength of paper or its resistance to picking.[2] It is not applicable to open or absorbent papers such as blotters or roofing felt. The waxes used in this test have graded adhesive powers so that when they are pulled from the sheet there is an increase in amount of pull administered to the surface fibers or coating as the wax numbers increase. The highest number of the wax in the series which does not disturb the surface of the paper is the numerical rating of pick.

NOTE.—Attention is called to the fact that, since the waxes specified are proprietary products the composition of which is not divulged, and since no method of calibrating the waxes has been accepted, this method is published for information only and use of it should be only as agreed upon by the parties concerned.

Apparatus and Material

2. The apparatus and material shall consist of the following:

(*a*) *Waxes.*—A proprietary series[3] of hard-resin, non-oily waxes numbered respectively from 2A to 32A. Each wax in the series is made up according to a particular formula and molded in stick form and is progressively more adhesive than that of the next lower one in the numerical series.

(*b*) *Heat Source.*—An alcohol or bunsen burner.

(*c*) *Wooden Block.*—A wooden block about $3\frac{1}{2}$ by $1\frac{1}{2}$ by $\frac{1}{4}$ in., having a hole $1\frac{1}{4}$ in. in diameter near one end.

Sampling

3. The paper shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585).[4]

Test Specimens

4. The test specimens shall be cut from the sample in such a way as to be thoroughly representative of it. The specimens may be of any convenient size.

Conditioning

5. The specimens shall be conditioned before testing in accordance with the Standard Method of Conditioning Paper

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products. Published as information, June, 1948.

[2] The following reference may be of interest in connection with this method:
William A. Kirkpatrick II, "The Use of Waxes and Case Testing Ink in Testing Printing Papers," *Paper Trade Journal*, Vol. 109, No. 12, September 21, 1939, pp. 36–38; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 22, No. 1, June, 1939, pp. 322–324.

[3] These waxes may be obtained from the Dennison Manufacturing Co., Framingham, Mass.

[4] 1946 Book of A.S.T.M. Standards, Part III-A.

and Paper Products for Testing (A.S.T.M. Designation: D 685).[4] Tests shall be made in an atmosphere conditioned in accordance with Method D 685.

Procedure

6. (*a*) Place the test specimen on a smooth surface. This should not be glass, metal, or other highly heat conductive material, nor should it be artificially cooled. A hardwood block or table top is satisfactory. The sheet to be tested should be separated from the block or table top by eight to ten sheets of paper.

(*b*) Select a wax stick, make certain that the end is clean and flat, and heat the end in an alcohol or low gas flame, rotating the stick slowly between the thumb and finger, until several drops of melted wax have fallen. (Do not let the wax catch fire.) Quickly place the melted end of the wax on the surface of the paper specimen, with firm but not undue pressure. Allow the wax to cool for 15 min. Place over the wax the wooden block having a hole $1\frac{1}{4}$ in. in diameter near one end. With the wax protruding through the hole of the block, press the block down firmly with one hand and with the other pull the wax from the sheet with a quick jerk at right angles to the paper surface. Examine both the tip of the wax and the specimen.

(*c*) Repeat the test if necessary, using the waxes in ascending numerical order until the surface of the paper specimen breaks, picks, or lifts. Make a separate test on each side of not less than five test specimens. On each test, record the highest numerical designation of the wax that does not disturb the surface of the paper, and average the results to the nearest whole number.

Report

7. (*a*) The average highest numerical designation of the wax that does not disturb the surface of the paper shall be reported as the surface pick test number. The report shall also include the number of tests made.

(*b*) In the case of uncoated paper, if it is possible to identify the two sides, results shall be reported separately for the felt side and the wire side.

NOTE.—The wire and felt sides of the paper may be determined by the Standard Methods of Test for Wire and Felt Sides of Paper (A.S.T.M. Designation: D 725).[4]

(*c*) On coated or machine-coated paper, the report shall also state whether the picking or lifting occurred on the coating or on the body stock or both. This shall be determined by examining the wax after removal from the paper being tested.

Precision

8. The surface pick test number obtained by this method should have a precision of approximately plus or minus one full number.

REPORT OF COMMITTEE D-7*

ON

WOOD

Committee D-7 on Wood held meetings at Atlantic City, N. J., on June 18, 1947, in conjunction with the Annual Meeting of the Society, and at Chicago, Ill., on March 16, 1948, at the time of the American Railway Engineering Association Convention.

Organization of the new Subcommittee VII on Poles and Cross Arms under the chairmanship of C. D. Hocker has been practically completed, with a membership now numbering twenty-four. A small task group has been appointed to work on one of the first major projects, namely, the draft of a tentative method of conducting static tests of full-sized wood poles. Substantial progress is expected during the coming year.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. J. Markwardt.

Honorary Chairman, Hermann von Schrenk.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Methods of Testing Small Clear Specimens of Timber (D 143 – 47 T) be approved for reference to letter ballot of the Society for adoption as standard without revision.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Wood Paving Blocks (W. H. O'Brien, chairman) reported that a review had been made of the Standard Specifications for Wooden Paving Blocks for Exposed Pavements (D 52 – 20) and that no revisions are recommended. Progress has been continued in the revision of the draft of a new specification for creosoted wood block flooring for interior use. This revision is now ready for submittal to the committee for approval. Consideration is being given by the subcommittee to the preparation of two additional specifications for wood block flooring for interior use: one for untreated blocks, and one for blocks treated with preservatives other than creosote.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman) reported substantial progress on the development of the new secondary standard for testing small clear specimens of wood, based on the use of smaller specimens than required in Standard Methods D 143. It is anticipated that this method will be presented for consideration as a new tentative during the coming year.

This report has been submitted to letter ballot of the committee, which consists of 52 members; 45 members returned their ballots, of whom 44 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

L. J. MARKWARDT,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE D-8*

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials held one meeting during the past year in Washington, D. C., on March 5, 1948.

The present officers of the committee have been re-elected for the ensuing term of two years.

In September, 1947, Mr. Lester Kirschbraun, a former member of Committee D-8, died in California. Mr. Kirschbraun had been a member of the Society since 1909 and was especially active in the affairs of Committee D-8, Committee D-4 on Road and Paving Materials, and Committee D-1 on Paint, Varnish, Lacquer, and Related Materials. A memorial resolution has been prepared by these three committees and a copy sent to his family.

Mr. Prevost Hubbard, who served Committee D-8 as secretary for 24 years, retired professionally on December 31, 1947, and resigned from the Society at the same time. His absence from Committee affairs will be keenly felt.

During the year the Advisory Committee created a new Subcommittee XIV on Compatibility Tests under the chairmanship of J. W. Donegan to develop tests for determining the compatibility of bituminous materials for roofing and waterproofing.

REVISION OF TENTATIVE

Committee D-8 joins with Committee D-4 in recommending that the Tentative Methods of Sampling Bituminous Materials (D 140 - 46 T) be revised by the addition of a new Section 1 on Scope which is given in detail in the Report of Committee D-4.[1] This revision was approved by Committee D-8 in June, 1947, but had not been concurred in by Committee D-4 at that time.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following tentatives be continued in their present status as they are all being actively studied with the probability that there will be revisions in each of them:

Tentative Specifications for:

Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 46 T),

Asphalt Roofing Surfaced with Mineral Granules (D 249 - 46 T),

Coal Tar Pitch for Steep Built-Up Roofs (D 654 - 42 T),

Tentative Recommended Practice for:

Accelerated Weathering Test of Bituminous Materials (D 529 - 39 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 65 voting members; 53 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. S. MILLER,
Chairman.

H. C. HOWELL,
Secretary.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] See p. 383.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE D-9*

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the Annual Meeting of the Society in June. These meetings were held in Atlantic City, N. J., on November 4, 5, and 6, 1947, and in Dayton, Ohio, on February 23, 24, and 25, 1948.

At these meetings actions were taken which resulted in several new methods, revisions in existing standards, and other changes in some of the standards for which Committee D-9 is responsible. Some projects which are still under way are mentioned later in this report.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. T. Hatcher.
Vice-Chairman, E. A. Snyder.
Recording Secretary,
O. E. Anderson.
Membership Secretary,
H. A. Eysenbach.

Subsequent to the 1947 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Natural Block Mica and Mica Films Suitable for Use in Fixed Mica Dielectric Capacitors (D 748 - 45 T), and
Thermosetting Materials (D 709 - 46 T).

Revision of Tentative Methods of:

Conditioning Plastics and Electrical Insulating Materials (D 618 - 46 T),
Testing Insulating Oil (D 117 - 46 T),
Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 46 T),
Measuring Dimensions of Rigid Rods Used in Electrical Insulation (D 741 - 43 T),
Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 - 46 T), and
Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 46 T).

Tentative Revision of Standard Methods of:

Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 46), and
Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (D 668 - 44).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the revised tentatives together with the tentative revisions appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, and also in the 1947 Compilation of A.S.T.M. Standards on Electrical Insulating Materials.

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is submitting one new tentative, revision of one standard with reversion to tentative, revisions in three tentatives, tentative revisions of two standards, adoption of two tentatives as standard, adoption as standard of a tentative revision of a standard, and the withdrawal of two tentatives.

These recommendations have been submitted to letter ballot of the committee and the results will be reported at the Annual Meeting.[1]

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Some of the projects now in progress are reviewed in the following summary of committee activities:

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (P. E. Demmler, chairman).—The reorganization of Subcommittee I was completed during the year. Sections on oxidizing, heat reactive, laminating, and high temperature insulating varnishes were organized to consider changes in the Tentative Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 46 T) or the preparation of additional methods, made necessary by recent developments in this field. Other sections were organized to study or develop the necessary test methods. Tentative Methods D 115 are to be rewritten to make them more useful for definition, classification, and control purposes.

A section on lac and lac products was reestablished.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (G. H. Mains, chairman) has had an active year. In cooperation with Committee D-20 on Plastics a revision of Tentative Specifications D 744 is being prepared which incorporates four electrical grades of nonrigid vinyl chloride plastics. It is also working on specifications for an electrical grade of cellulose acetate film, and is reviewing this in view of some negative ballots. The subcommittee has had requests for electrical specifications for polyethylene, nylon, and tetrafluoroethylene at ultra-high frequencies, but test methods and equipment are still lacking. An extensive revision of the Tentative Specifications for Phenolic Molding Compounds (D 700 - 45 T) has been prepared and should be ready for committee letter ballot shortly.

The cooperative work with Subcommittee XII on methods for measuring insulation resistance of sheet materials has demonstrated that the present tapered pin methods are inadequate in many cases. A new method has been developed which offers promise in separating the body and surface components of insulation resistance. In cooperation with Subcommittee I of Committee D-20 a radical improvement in the tension test method for rods and tubes has been worked out and is being submitted as a revision of the Tentative Method of Test for Tensile Properties of Plastics (D 638 - 46 T).

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Section A is cooperating with Committee D-2 on Petroleum Products and Lubricants in the investigation of the acid and base number test to determine its suitability for transformer oils.

Section B has been primarily concerned with a continuous study of the behavior of new and reclaimed transformer oils in actual transformer service. The program initiated in 1945[2] is being continued. This includes the testing of oil samples drawn from transformers in operation at plants of the American Gas and Electric Co., Commonwealth Edison Co., Detroit Edison Co., and New England Power Co. Sufficient data have not been accumulated to warrant final conclusion.

Section B has also accepted the study of inhibited oil for which purpose a subsection has been organized to consider the test methods best adapted for this study.

Section D on Saponification Number had very little work to do last year. There is still considerable skepticism in the minds of many of the members of Subcommittee IV as to whether the saponification test has any significance when applied to the testing of electrical insulating oils. The section pointed out to Subcommittee IV that Subcommittee XIII of Committee D-2, as a result of a cooperative testing program, had approved the Method of Test for Saponification Number of Petroleum Products by Potentiometric Titration (D 939 - 47 T) for publication as tentative. The scope of this method indicates that no relationship exists between the colorimetric and electrometric saponification number.

---

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 275 (1945).

Section E developed the Tentative Method for Sampling Electrical Insulating Oils (D 923 - 47 T) which was accepted as tentative by the Society during the past year. Some further work is to be done in developing a special method of sampling oils for absolute moisture determination. Procedure in this connection is pending the results of some round-robin tests now in progress to check reproducibility of moisture determinations with ordinary sampling methods.

Section F is continuing its investigation of test methods used by cable manufacturers to evaluate the stability of cable oils. At present they are comparing test results on power factor as obtained by each manufacturer's individual method with the results obtained when tests are run on oil aged for 96 hr. at 100 C. This work will be continued in an endeavor to standardize a test procedure which will be acceptable to all cable manufacturers.

During the current year, Section I has considered the use of this same procedure for determining the power factor of askarels. Although little experience is available on the testing of power factor of askarel, it appears that the method will be satisfactory provided a note is added to the procedure that phenolic insulation should not be used where it may come in contact with the askarel, as it causes contamination. Such a note will be prepared and submitted to Subcommittee IV for action.

Section J recommended that the Tentative Methods of Test for Gas Content of Insulating Oils (D 831 - 45 T) be approved for adoption as standard.

Subcommittee IV voted to recommend the Tentative Methods of Test for Dielectric Strength of Electrical Insulating Oils of Petroleum Origin (D 877 - 46 T) for adoption as standard.

Investigation conducted during the year by Section L has determined that Method D 877 is satisfactory for determining the dielectric strength of askarels but the section is still studying the modifications necessary to make the method applicable for use in testing the dielectric strength of heavy viscous oils.

Statements on definition and significance of tests for free sulfur and saponification numbers were developed by Section M, approved by Subcommittee IV, and submitted to Subcommittee XI for approval.

Work is still continuing on the development of satisfactory statements covering definition and significance of the tests for power factor and gas content.

In Section N, further testing by the cooperating laboratories of samples of insulating oils has shown that there is still considerable variation in test results using the methods available. These variations are attributed to water retention by the sample bottles, faulty end point in titration methods, and failure of the methods to detect only water. Work is continuing to overcome these difficulties by improvement in technique of handling and analyzing samples.

Section O on Askarels is working on slight modifications necessary to make Tentative Method D 924 on power factor suitable for use on askarels.

In the reorganization of the test methods, the subcommittee has now developed tests and statements for free sulfur and power factor. These methods will be issued under their own A.S.T.M. designation and references to them will be inserted in the proper places in Tentative Method D 117. After their acceptance by letter ballot of Committee D-9 during the coming year, Tentative Method D 117 will be recommended for adoption as standard. Upon approval of this action the present Standard D 117 will be withdrawn.

Section S completed its work on revision of the Method of Test for Detection of Free Sulfur in Electrical Insulating Oils which is submitted for publication as tentative. The section is at present working on the development of a new test method which will determine the presence of free sulfur as detected by the proposed tentative method. In addition, it will also detect the presence of combined sulfur compounds which under certain service conditions might be changed into corrosive sulfur potentially dangerous to electrical apparatus. After final development of this test method and after sufficient experience has been obtained by its use, it is hoped that it may prove to fulfill all the requirements of a sulfur test permitting withdrawal of the one now offered as tentative.

Section T on Interfacial Tension Test conducted round-robin test No. 2 using oils of high and low interfacial tension. The results showed surprisingly good agreement between the cooperating laboratories. It was decided to conduct another round-robin test using oils of an intermediate interfacial tension. On the basis of the results obtained from both tests, the reproducibility and repeatability of the method will be determined. During the year an adaptor was developed by the section chairman for manual alignment of the tensiometer ring. The design has been submitted to the manufacturer for his consideration.

During the year Section X on Specifications adopted the following tests and specification limits of the test for use in a screening specification:

(*1*) Dielectric strength, not less than 26 kv.

(*2*) Neutralization number, not more than 0.05 mg. KOH/g.

(*3*) Free sulfur, must pass A.S.T.M. test.

(*4*) Viscosity, not more than 65 sec. Saybolt Universal at 100 F.

(*5*) Pour point, not higher than −40 F.

(*6*) Flash point, not less than 265 F. open cup.

These specifications will be included in a recommended practice specification which the subcommittee is developing as a guide for the purchase of transformer and circuit-breaker oils.

The second of a series of symposia on insulating oil sponsored by Subcommittee IV was held at the Annual Meeting in Atlantic City, N. J., in June, 1947. The attendance at this symposium indicated the wide interest in the subjects of inhibited insulating oils and the meaning of power factor testing of oil, the chief subjects discussed in four papers presented. All of the papers and complete discussion were subsequently published in the ASTM BULLETIN, December, 1947. The committee is now preparing a program for its third symposium on insulating oil to be held at the 1949 spring meeting of Committee D-9.

Subcommittee V on Ceramic Products (K. G. Coutlee, chairman).—Section C on Steatite and Other Special Purpose Ceramic Bodies has been concentrating on the development of suitable test methods for evaluating moisture sensitivity of electrical properties of steatite on which progress can be reported. Similar studies are being conducted on glass-bonded mica by Section D and proposed test methods for this material are under consideration.

Subcommittee VI on Solid Filling and Treating Compounds (W. A. Evans, chairman) was rather inactive for several years. The former chairman, R. H. Titley, endeavored to revive its activities and through a questionnaire he obtained the ideas and interests of the members and industry. With this background the committee is organizing a project on insulating waxes and treating compounds in which there is a great deal of interest. This work will result in the revision of the Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 44) and the development of new and additional test methods.

Subcommittee VII on Insulating Fabrics (R. W. Chadbourn, chairman).—During the year the subcommittee approved the slow-rate-of-rise dielectric strength test as an alternate to the existing step-by-step test for varnished cloth and referred the changes in Standard Methods D 149 to make this a general test method, to Subcommittee XII for final review and approval. No objections to the new method have been received to date.

Data on the effect of 12 per cent elongation on the dielectric breakdown of tapes continues to be accumulated slowly, and it appears that the subcommittee will soon be able to set specification limits, at least for the thicker tapes, which show more consistent breakdowns than the thinner tapes. The subcommittee is also reviewing the specification limits of 6 per cent elongation, as recent data indicates that for present-day tapes the limits may be a little high.

During the year a round-robin study was made of the effect of exposure to relative humidities of 90 and 96 per cent on the dielectric strength of varnished cloth tapes. The results of this investigation showed that the dielectric strength was on the average about a third less at 96 than at 90 per cent. Because of this and because tapes are frequently exposed to practically saturated atmospheres, it was voted to request Subcommittee X to consider the adoption of 96 per cent as an additional standard relative humidity.

The section on Treated Sleeving is beginning a round-robin study of the effect of hot oil on treated sleeving. Statements of significance of tests for treated sleeving, covering conditioning, dimensions, dielectric strength, aging test, heat and endurance, and rate of burning were accepted by Subcommittee XI with minor revisions and reapproved by Subcommittee VII. A few additional statements of significance of test for varnished cloth and cloth tapes are in process of preparation. The Section on Treated Glass Fabrics has begun a round-robin investigation to determine the effect of heat on the flexibility of treated glass tapes. The problem of making dielectric strength tests at elevated temperatures is also being studied.

The Section on Electrical Adhesive Tapes has completed its preparation of test methods for conditioning, thickness, breaking strength, dielectric strength, adhesion, storage stability, and electrolytic corrosion by the conductance method. An alternate method for electrolytic corrosion, involving a tension test, is receiving further study. The problem of the effect of solvents and varnishes on the tapes is also under consideration.

Subcommittee VIII on Insulating Papers (C. T. Hatcher, chairman).—Work has been carried on covering the development of a conductivity method for insulating papers and a test for water and alcohol soluble matter. Consideration is being given to revising the acidity or alkalinity method so as to avoid unnecessary repetition and to effect simplification of Methods D 202. The pH method in D 202 is being studied to determine if it can be revised so as to permit appropriate references to the

Tentative Method for pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T). This work is being carried on in conjunction with the Joint D-9-D-6 Committee.

There appears to be a need for an A.S.T.M. procedure for determining chlorides in paper. Approval for this work has been granted by Committee D-9 and the Joint D-6-D-9 Committee has been requested to proceed with this work.

The rate of impregnation test in Method D 202 is being revised[3] so as to incorporate changes which have been made in the design of the penetrometer. The title will be changed from "Rate of Impregnation" to "Impregnation Time."

Consideration is being given to the acceptance of a Tentative Method of Test for Stretch of Paper and Paper Products Under Tension which has been prepared by Committee D-6 on Paper and Paper Products.[4]

The section which was formed to prepare specifications for insulating papers has been actively engaged in the preparation of a specification for absorbent insulating paper. Representation was obtained on the section from both producer and consumer interests. Several drafts of the specification have been prepared and it is expected that a specification will be ready for approval during the coming year. Some development work is being carried on to establish limits for the tests which will be specified. Upon completion of this specification, the section will prepare specifications for other types of insulating papers.

Subcommittee IX on Mica Products (E. O. Hausmann, chairman) has had under consideration sampling methods for addition to Specifications D 748. Further improvement of these specifications and the development of a specification for raw mica for other than dielectric capacitor use are being considered.

Much time and effort has been spent in the development of colored photographic transparencies. It now has been determined that duplicate transparencies taken from the originals will not satisfactorily show the various mica imperfections as required. Also it is known that the transparencies must be colored rather than black and white. The subcommittee is now proceeding with the necessary details preliminary to the establishment of secondary colored photographic transparencies. It is hoped that this project is now on the way toward satisfactory completion. Needless to say, these transparencies will greatly enhance the value of our visual classification system for mica.

Subcommittee X on Conditioning (M. L. Macht, chairman) serves both Committees D-9 and D-20 on matters referring to conditioning.

A further revision of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 47 T) was approved last year. In this revision the scope has been amplified in order to outline more clearly the nature of the method; two temperatures, 130 C. and 190 C., have been added to standard test temperatures; Section 3 on conditioning prior to test was revised to eliminate a differentiation between a standard and a functional procedure and also it has been enlarged to include three further procedures, known as Procedure C, 90 per cent relative humidity at a temperature of 35 C. (95 F.); Procedure D, immersion in distilled water for 24 ± $\frac{1}{2}$ hr. at 25 ± 1 C. (77 ± 1.8 F.); and Procedure E, immersion in distilled water for 48 ± $\frac{1}{2}$ hr. at 50 ± 1 C. (122 ± 1.8 F.).

The work load of this subcommittee has increased tremendously because of various proposed changes in standard conditions by Government laboratories, military forces, and electrical groups. Consequently, two sections have been established. Section A will handle revisions of Methods D 618 when requested, and Section B has been appointed for the purpose of developing high humidity techniques and to standardize methods of measuring humidity.

---

[3] See Editorial Note, p. 409.

[4] 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

Subcommittee XI on Significance of Tests (J. H. Adams, chairman) has reviewed and approved statements as to the significance of certain tests applied to electrical insulating oils submitted by Subcommittee IV, and statements as to the significance of tests applied to flexible treated sleeving used for electrical insulation submitted by Subcommittee VII.

Much of the time and thought of Subcommittee XI has been directed to the completion of a "Recommended Practice for Writing Statements as to the Usefulness of Tests of Electrical Insulating Material". Understanding as to what properly should be included in such statements has seemed very desirable for the guidance of authors and reviewers. The recommended practice was approved by letter ballot of the subcommittee and submitted to Committee D-9 for letter ballot approval. Thought is being given to encouraging the writing of statements of test utility for test methods whose utility has not been defined.

Subcommittee XII on Electrical Tests (K. N. Mathes, chairman).—During the past year Section A on Insulation Resistance held several all day meetings and has nearly completed a comprehensive revision of the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 46). Section A has also completed a round-robin test on pin-type samples for resistance measurements and concluded that such samples usually emphasize surface resistance rather than volume resistance. In consequence, Subcommittee XII has recommended that the use of pin-type samples be discontinued. A paper describing the reasons for this recommendation and suggestions for raw methods will be prepared for publication.

Section C on Dissipation Factor has made a list of laboratories which have equipment to make measurements up to 30,000 mg. This equipment has been cataloged and plans made for round-robin measurements.

Section E on Dielectric Strength has proposed a slow rate of voltage rise as an alternate method to the step-by-step method for dielectric strength. This proposal has been accepted for letter ballot by Committee D-9.

Section F on Arc Resistance has held several all day meetings and has completed the comprehensive revision of the Standard Method of Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 – 42). In this revised method changes have been made in the testing electrodes, and circuit constants have been specified within close limits. Proposals for additional work to develop new methods for arc resistance have been made.

A new Section H on Electrodes has been formed with K. G. Coutlee as chairman. This section has been asked to investigate materials, methods of application, and characteristics of electrodes for all types of electrical measurement.

This report has been submitted to letter ballot of the committee, which consists of 139 voting members; 69 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. W. ORR, *Chairman.*

GORDON THOMPSON, *Vice-Chairman.*

W. A. ZINZOW,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:

Testing Pressure Sensitive Adhesive Tapes Used in Electrical Insulation (D 1000 - 48 T).

Revision of Tentative Methods of:

Sampling and Testing Untreated Papers Used in Electrical Insulation (D 202 - 47 T).

Tentative Revision of Standard Methods of:

Testing Pasted Mica Used in Electrical Insulation (D 352 - 39).

These recommendations were accepted by the Standards Committee on December 22, 1948, and the new and revised tentatives together with the tentative revision of D 352 appear in the 1948 Supplement to Book of A.S.-T.M. Standards, Part III-B.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentatives covering electrical insulating materials which are referred to earlier in this report. The standards and tentatives appear in their present form either in the 1946 Book of A.S.T.M. Standards Part III-B, or the 1947 Supplement Part III-B as indicated by the final number in the A.S.T.M. designation.

NEW TENTATIVES

Tentative Method of Test for Detection of Free Sulfur in Electrical Insulating Oils:

This method has been developed for the purpose of detecting the presence of free sulfur in electrical insulating oils. It is recommended that this method, as appended hereto,[1] be accepted for publication as tentative.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Tentative Method of Test for High Voltage, Low Current Arc Resistance of Solid Electrical Insulating Materials (D 495 - 48 T):

This method of test is a complete revision of the current Standard Method D 495 - 42 resulting from a very complete study and comprehensive survey of test results which has been carried on for some time. It is recommended that this method, as appended hereto,[2] be accepted for publication as tentative.

REVISIONS OF TENTATIVES

Tentative Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 46 T):

These revisions involve some slight change in procedure for certain of the tests, the purpose of which is to clarify the procedures involved. They also include the deletion of two types of tests, namely: volatile matter by volume and dielectric strength of varnish in the liquid state, which have been found by experience to have little, if any, significance and value.

Section 1.—In the list of tests, change "Oil Proof Test" to read "Oil Resistance."

Section 2.—Add the following as a new Paragraph (*c*) and reletter the subsequent paragraphs accordingly:

> (*c*) All varnish films for tests shall be prepared at 25 C. and 50 per cent relative humidity, or such other temperature and humidity conditions as may be adopted by Committee D-9 for such purposes, and the air of the room shall be freed of dust by some satisfactory method of filtering.

In the present paragraph (*c*), change the third sentence to read as follows: "It shall be such that when the strips are dipped in the varnish at a temperature of approximately 25 C. (77 F.) and are withdrawn slowly and uniformly at the rate of 4 in. per min. the average thickness of the film remaining on each side of a strip when dry shall be 0.0010."

---

[1] This method was accepted by the Society with revisions as given in the Summary of Proceedings on p. 24 and the revised method appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

[2] This revised method was accepted by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

Sections 17 and 18.—Change the title of these two sections from "Heat Endurance Test" to read "Heat Flexibility Test."

Section 19.—Change the fifth sentence to read as follows: "The specimens shall be wiped with cotton batting, and the effect of the oil should be based on whether the varnish film dissolved, blistered, wrinkled, loosened, or was not visibly affected physically."

Also, delete Item *(3)* and the note referring to discoloration of the oil.

Sections 25 to 32.—Delete these sections on Volatile Matter by Volume and Dielectric Strength of Varnish in the Liquid State.

Tentative Specifications for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 – 47 T):

This revision consists of the addition of Rockwell hardness requirements for sheet grades and dimensional tolerances for cut lengths of tubes. These are clarifications and extensions of present specifications:

Table II.—Add the following Rockwell hardness requirements to this table:

Grade	Rockwell hardness, min.
Bone	R-80
Commercial	R-50
Electrical	R-50

Section 10.—Add the following as a new Paragraph *(c)*:

(c) The maximum permissible variations in lengths of circular sawed pieces of rods shall be as follows:

Nominal Length, in.	Permissible Variations in Length as Cut, plus or minus, in.
0 to 3, incl.	0.015
Over 3 to 6, incl.	0.020
Over 6 to 12, incl.	0.030
Over 12 to 24, incl.	0.035
Over 24 to 48, incl.	0.040
Over 48 to 84, incl.	0.062

Section 11.—Add the following as a new Paragraph *(c)*:

(c) The maximum permissible variations in length of circular sawed pieces of tubes shall be as follows:

Nominal Length, in.	Permissible Variations in Length as Cut, plus or minus, in. Diameter 0 to 3	0 to 6
0 to 3, incl.	0.015	0.020
Over 3 to 6, incl.	0.020	0.025
Over 6 to 12, incl.	0.030	0.035
Over 12	0.035	0.045

Tentative Specifications for Laminated Thermosetting Materials (D 709 – 47 T):

This revision consists of the addition of four glass-base grades of materials together with the specified requirements and tolerances. This is an extension of the specifications to cover newly developed materials of this type.

Section 2.—Add the following requirements for the type IV, glass-base laminated material:

Type IV. Glass-base laminated material:
Grade B1. Staple-fiber-type glass cloth; general-purpose, heat-resisting,
Grade G2. Staple-fiber-type glass cloth; electrical and heat-resisting,
Grade G3. Continuous-filament-type glass cloth; general-purpose, and
Grade G5. Continuous-filament-type glass cloth, melamine binder; general-purpose. Good arc and flame resistance.

Table X.—Make the following revisions in this table as shown in the accompanying Table I.

Appendix.—Add the descriptions of the new type IV grades of glass-base laminated material as given above in Section 2.

TENTATIVE REVISIONS OF STANDARDS

Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 45):

The revision of this standard consists of six statements for definition and significance of six properties of flexible treated sleeving covered by the method. The proposed revision, appended hereto,[3] is recommended for publication as tentative.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF SHEETS.[a]

Nominal Thickness, In.	Grades							
	X, P, XX, XXP, XXX, XXXP, A[b]	C	CE	L	LE	AA	G1, G2	G3, G5
	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus
0.010	0.002	....	....	0.003	....	....	....	....
0.015	0.0025	....	....	0.0035	0.0035	....	....	....
0.020	0.003	....	....	0.004	0.004	....	....	....
0.025	0.0035	....	....	0.0045	0.0045	....	....	....
1/32	0.0035	0.0065	0.0065	0.005	0.005	....	0.008	0.0065
3/64	0.0045	0.0075	0.0075	0.0055	0.0055	....	0.010	0.0075
1/16	0.005	0.0075	0.0075	0.006	0.006	0.018	0.010	0.0075
3/32	0.007	0.009	0.009	0.007	0.007	0.0018	0.012	0.009
1/8	0.008	0.010	0.010	0.008	0.008	0.020	0.012	0.012
5/32	0.009	0.011	0.011	0.009	0.009	....	0.015	0.015
3/16	0.010	0.0125	0.0125	0.010	0.010	0.024	0.019	0.019
7/32	0.011	0.014	0.014	0.011	0.011	....	0.021	0.021
	Plus or Minus	Plus Only	Plus or Minus	Plus Only	Plus or Minus	Plus or Minus	Plus or Minus	Plus or Minus
1/4	0.012	0.030	0.015	0.024	0.012	0.028	0.022	0.022
5/16	0.0145	0.035	0.0175	0.029	0.0145	0.034	0.026	0.026
3/8	0.017	0.040	0.020	0.034	0.017	0.038	0.030	0.030
7/16	0.019	0.044	0.022	0.038	0.019	0.044	0.033	0.033
1/2	0.021	0.048	0.024	0.042	0.021	0.048	0.036	0.036
5/8	0.024	0.053	0.027	0.048	0.024	0.058	0.040	0.040
3/4	0.027	0.058	0.029	0.054	0.027	0.068	0.043	0.043
7/8	0.030	0.062	0.031	0.060	0.030	0.076	0.046	0.046
1	0.033	0.065	0.033	0.065	0.033	0.086	0.049	0.049
1 1/8	0.035	0.069	0.035	0.069	0.035	....	0.053	0.053
1 1/4	0.037	0.073	0.037	0.073	0.037	0.106	0.055	0.055
1 3/8	0.039	0.077	0.039	0.077	0.039	....	0.058	0.058
1 1/2	0.041	0.081	0.041	0.081	0.041	0.124	0.061	0.061
1 5/8	0.043	0.085	0.043	0.085	0.043	....	0.064	0.064
1 3/4	0.045	0.089	0.045	0.089	0.045	0.144	0.067	0.067
1 7/8	0.047	0.093	0.047	0.093	0.047	....	0.070	0.070
2	0.049	0.097	0.049	0.097	0.049	0.160	0.073	0.073

[a] On sheets of nominal thickness not listed in this table, the permissible variations shall be the same as for the next greater thickness.

[b] The minimum thickness for grades XXP, XXX, and XXXP is 0.015 in., and for grade A is 0.025 in. Grades P, XXP, and XXXP are not available in thicknesses over 1/4 in.

Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 44):

This revision consists of the addition of a slow-rate-of-rise test. This method of test is coming into use in commercial laboratories and has been checked and found to be useful and informative. The proposed tentative revision is as follows:

Section 2 (c).—Change the second sentence to read as follows: "For fairly rapid determinations, but laying more emphasis on the time factor, a choice of two tests is offered: (*1*) A step-by-step test, or (*2*) an equivalent slow-rate-of-rise test."

Section 10.—Add the following as a new Paragraph (*c*):

(*c*) *Slow-Rate-of-Rise Test*.—An initial voltage shall be applied equal to approximately 50 per cent of the breakdown voltage in the short-time test, or as specified in the material specifications. The voltage shall then be increased at a uniform rate, as stated in the various material specifications, up to the point of breakdown. The specified rate is chosen to give approximately the same voltage-time exposure of the test specimen as provided in the step-by-step test.

---

[3] This tentative revision was accepted by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

Appendix.—Insert a new paragraph after the second paragraph, to read as follows:

To accommodate the wide range of speed control required by the short-time and slow-rate-of-rise tests, the apparatus is equipped with a gear shift. This is so designed that with the gears in one position, short-time tests may be made; shifting the gears by means of the knob, *D*, Fig. 1, adapts the apparatus to slow-rate-of-rise tests.

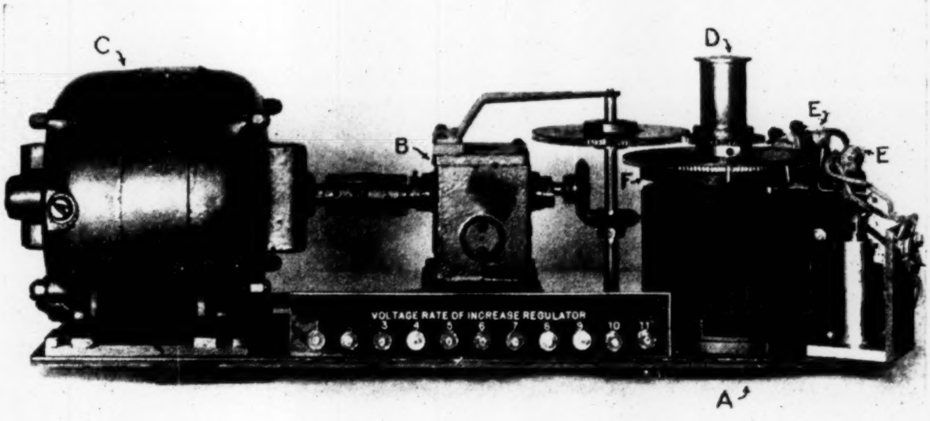


FIG. 1.—Variable Auto-Transformer, Motor Controlled, with Automatic Limit Features

Figure 1.—Substitute the accompanying Fig. 1 for the present figure.

Change the last paragraph to read as follows:

Over-travel of the motor is prevented by mechanical tripping of small mercury switches, *EE*, Fig. 1, in the control circuit. Tripping is accomplished by pins which may be inserted into various holes, as desired, around the edge of a disk, *F*, mounted on the Auto-transformer rotor. One switch is connected in the forward circuit, and the tripping pin located so as to open the switch when maximum voltage is reached. The other switch is connected in the reversing circuit, and its tripping pin may be placed so as to open the circuit at the point of zero voltage, or at a higher point, if desired. Thus, for slow-rate-of-rise tests, the pin would be set to trip the switch when the rotor returned to the position of the proper starting voltage.

Add a new paragraph to read as follows:

For convenience and ease of operation, the control and supply circuits may be wired to trip simultaneously at the instant of breakdown, but the indicating voltmeter may be so wired that it continues to read the breakdown voltage, after the control and supply circuits are tripped, until the rotor is reversed and returned to the starting position. This should only be done if the characteristics of the voltmeter and circuit used are such that an error excessive for the type of test being performed would not thereby result. The additional error introduced by the voltmeter and this type of connection should not exceed 3 to 5 per cent with the most suitable types of instruments.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:
Tensile Strength of Molded Electrical Insulating Material (D 651 – 42 T), and
Gas Content of Insulating Oils (D 831 – 45 T).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 45):

The committee recommends that the tentative revision[4] of these methods involving Section 14(*a*) and changes in Table I submitted in December, 1944, be approved for reference to letter ballot of the Society for adoption as standard.

WITHDRAWAL OF TENTATIVES

Tentative Methods of Test for Compressive Strength of Electrical Insulating Materials (D 649 – 42 T), and for Flexural Strength of Electrical Insulating Materials (D 650 – 42 T):

The committee recommends that these tentatives be withdrawn because they have been superseded by other test methods developed under the joint jurisdiction of Committees D-9 and D-20 which are applicable to plastics and electrical insulating materials.

---

[4] 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

REPORT OF COMMITTEE D-10*

ON

SHIPPING CONTAINERS

Committee D-10 on Shipping Containers held two meetings during the past year. The committee is now following the practice of holding two meetings annually, one in the east and one in the west. The fall meeting was held in Chicago, Ill., at the Quartermaster Food and Container Institute on October 31, 1947, and was preceded by a full day of subcommittee meetings. There were 32 members and visitors who attended this meeting. The custom of presenting a technical paper at the meeting was instituted with A. V. Grundy discussing "Standards Required for Quartermaster Packing." The spring meeting was held on April 27, 1948, at Cleveland, Ohio, and it also was preceded by a full day of subcommittee meetings. There were 47 members and visitors in attendance. Following the custom begun at the fall meeting, R. A. Mantz spoke on "Research in Container Design and its Relation to Materials Handling."

All six of the subcommittees of Committee D-14 met at both the fall and spring meetings. The attendance at these sessions was large and representative, and the result of this activity is evidenced by the new tentatives under consideration and the formulation of definite programs for future work.

At the present time, the committee consists of 75 members, of whom 26 are classified as producers, 27 as consumers, and 22 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, T. A. Carlson
Vice-Chairman, R. C. McKee
Secretary, E. R. Stivers

Subcommittee II on Methods of Testing (E. R. Stivers, chairman) has completed a new method for vibration testing which will be submitted to the Administrative Committee on Standards in the near future for publication as tentative.[1]

RECOMMENDATIONS ACCEPTED BY ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Water Resistance of Containers by the Spray Method. The new tentative method was accepted by the Standards Committee on November 6, 1948, and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, bearing the designation D 951 – 47 T.

On April 6, 1948, the Administrative Committee on Standards accepted the Tentative Method of Drop Test for Bags, which has been issued under the designation D 959 – 48 T.[2]

This report has been submitted to

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] See Editorial Note, p. 416.

[2] 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

letter ballot of the committee, which consists of 75 members; 55 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. CARLSON,
Chairman.

EARL R. STIVERS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the recommendation that the Definitions of Terms Relating to Shipping Containers be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on August 3, 1948, and the new tentative definitions appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B, bearing the designation D 996 – 48 T.

On September 29, 1948, the Administrative Committee on Standards accepted the following recommendations submitted by Committee D-10:

New Tentative Method of:

Drop Test for Cylindrical Shipping Containers (D 997 – 48 T), and
Vibration Test for Shipping Containers (D 999 – 48 T).

The new tentatives appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

The proposed Tentative Method of Test for Penetration of Liquids Into Submerged Containers was accepted as tentative by the Standards Committee on November 15, 1948. The new tentative method has been assigned the designation D 998 – 48 T, and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

REPORT OF COMMITTEE D-11*

ON

RUBBER AND RUBBER-LIKE MATERIALS

Committee D-11 on Rubber and Rubber-Like Materials held one meeting during the year in Washington, D. C., on March 5, 1948, in connection with the Spring Group meetings of A.S.T.M. committees. The subcommittees, however, have in many cases met separately and have carried on a great deal of activity by correspondence.

At the spring meeting, the committee unanimously elected four honorary members in recognition of their long service and many valuable contributions to the work of the committee. These honorary members are A. H. Flower, W. L. Sturtevant, H. S. Vassar, and P. L. Wormeley. It is hoped that they will continue to guide and advise the committee from their wealth of experience even though now retired from active business.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Simon Collier.
Vice-Chairman, H. G. Bimmerman.
Secretary, Arthur W. Carpenter.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

In the 1947 Annual Report of Committee D-11[1], reversion to tentative of the Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 39) was recommended and revisions were proposed. Since it was not possible to preprint the revised method at that time, the revisions were presented to the Society through the Administrative Committee on Standards subsequent to the Annual Meeting and were approved on November 6, 1947. This revised method appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B. Subsequently, a number of other recommendations listed below were also presented to the Society through the Standards Committee and were approved on February 6, 1948. These new and revised standards as well as the tentative revision appear in the 1948 Compilation of A.S.T.M. Standards on Rubber and Rubber-Like Materials:

New Tentative Methods of:

Test for Mechanical Properties of Elastomeric Vulcanizates under Compressive or Shear Strains by the Mechanical Oscillograph (D 945 – 48 T).

Revision of Tentative Methods of:

Testing Hard Rubber Products (D 530 – 44 T),
Testing Automotive Hydraulic Brake Hose (D 571 – 44 T).

Revision of Tentative Specifications for:

Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 – 46 T).

Tentative Revision of:

Standard Method of Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (D 531 – 41).

Withdrawal of Tentative Specifications for:

GR-S (Synthetic Rubber) Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is not Required (D 812 – 44 T).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 379 (1947).

The new tentative method of test covering the use of the Yerzley oscillograph is widely employed for the evaluation of the mechanical properties of elastomeric vulcanizates which find application in vibration damping service. The apparatus has been in extensive use for some time but no standardized procedure for it has previously been available. The revisions in Tentative Methods D 530 improved procedure details in the tension, cold flow, and softening point tests. In the case of the revisions of Tentative Methods D 571 a salt spray test for the metal end fittings of the hose has been added and the whip test procedure has been revised to cover the testing of longer lengths of hose which are coming into use. The revision of Tentative Specifications D 735 added a new suffix letter "R" to the code system used for specifying grades. This letter will be employed to signify that a resilience test is required. The tentative revision of Standard Method D 531 makes provision for the use of a larger ball point on the instrument which is necessary when testing very soft stock. Tentative Specifications D 812 were withdrawn because there is no longer any need for this grade of sheath of relatively low abrasion resistance.

NEW TENTATIVE

Tentative Method of Test for Volume Resistivity of Electrically Conductive Rubber and Rubber-Like Materials.[2]—This method of test was published as a proposed method for information in the February, 1948, Compilation of A.S.T.M. Standards on Rubber and Rubber-Like Materials. Electrically conductive rubber finds important use in many services where development of static charges are serious and where they constitute a great safety hazard as is the case in the use of rubber in connection with the manufacture of explosives or in the handling of combustible materials. Reliable methods are needed for determining the conductivity of such rubber and its efficacy in the elimination of the accumulation of static. The proposed method has been thoroughly studied and evaluated by a special committee organized within the SAE-ASTM Technical Committee on Automotive Rubber and is believed to provide the best means now available for conducting this measurement.

REVISION OF TENTATIVES

The committee recommends that the following five tentatives be revised as indicated and continued as tentative:

Tentative Specifications for Friction Tape for General Use for Electrical Purposes (**D 69 - 46 T**).[3]—The following revisions will provide for a better quality of tape than is required under the present specifications. Tape of this quality which is more nearly like that furnished prior to the war is now being manufactured.

Section 5.—Designate the present paragraph as (*a*) and add the following Paragraphs (*b*) and (*c*):

> (*b*) The rate of separation between layers of the tape, when tested in accordance with the procedure described in Section 20 (*a*), shall not exceed 15 in. per min.
>
> (*c*) The rate of separation between layers of the tape, when tested in accordance with the procedure described in Section 20 (*b*), shall not exceed 15 in. per min.

Section 6.—Change to read as follows:

> 6. The tape shall show tackiness, that is, ability to stick to itself after light contact has been made, in the following combinations: (*1*) front to front, (*2*) back to back, and (*3*) back to front when tested in accordance with the procedure described in Section 21.

Section 8.—Add the following sentence to the end of this section: "The test for pinholes shall be made in accordance with the procedure described in Section 23."

---

[2] This method was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

[3] 1946 Book of A.S.T.M. Standards, Part III-B.

Section 10.—Add the following at the end of the first sentence: "in accordance with the procedure described in Section 24."

Section 12.—Add the following sentence to the end of this section: "Measurement of thickness and width shall be made in accordance with the procedure described in Section 26."

Section 17 (a).—Change the period of acceptance from "four" to "eight" months and the reference to Section 20 (*a*) instead of Section 20.

Section 20.—Add the following as a new Paragraph (*b*), relettering the present (*b*) as Paragraph (*c*):

> (*b*) *Oven Test.*—A tape specimen similar to that described in Paragraph (*a*) shall be exposed to dry air at a temperature of not less than 99 C. (210.2 F.) nor more than 101 C. (213.8 F.) for a period of 16 hr. The specimen shall be supported in the oven by means of clips or other suitable devices attached to the ends in such a way that the adhesion test portion, 19 in. in length, hangs free and not in contact with oven parts or other tape specimens. After a rest period at room temperature of not less than 4 hr. nor more than 8 hr., during which the aged specimen shall be protected from contact with foreign material, the specimen shall withstand the test prescribed in Paragraph (*a*), except that the weight applied to unwind the tape shall be 3 lb. per in. of width in place of 4 lb. per in. of width.

Section 21 (c).—Change to read as follows:

> (*c*) The two specimens shall then be picked up from the end last in contact with the roller using two pairs of tweezers, one for each specimen. Tackiness in at least two of the combinations (*1*), (*2*), and (*3*) specified in Section 6 shall be such that the point of separation remains approximately in the same horizontal position as the tweezers that pull the specimens apart. In the back to back combination the specimens shall show some indication of tackiness by the point of separation traveling along the tapes as the tweezers pull the tape apart. In pulling the specimens apart, one hand shall be kept in contact with a rigid support such as the edge of a table and the other hand slowly moved along the edge of the table to bring about the separation of the specimens.
>
> The rate at which the specimens are separated shall be steady and shall not exceed 1 in. per sec. The last 1 in. of specimen shall be disregarded.

Section 21 (d).—Delete this paragraph.

Tentative Specifications for Rubber Insulating Tape (**D 119 - 46 T**)[3]—The following revisions are recommended for the same reason as given above under Specifications D 69:

Section 2.—Change the first sentence to read as follows: "The tape shall consist of an unvulcanized or partially vulcanized natural rubber insulating compound which shall contain not more than 0.5 per cent of free sulfur by weight of the original compound when tested in accordance with the procedure described in Section 22."

Section 4.—Delete this section, renumbering the subsequent sections accordingly.

Section 5 (a).—Renumber as "4" and change to read as follows:

> 4. (*a*) The tensile strength of the tape, when tested in accordance with the procedure described in Section 17, shall be not less than 250 psi.

Section 5 (b).—Change the percentage of elongation from "250" to read "300" per cent.

Section 6.—Renumber as "5" and change to read as follows:

> 5. The dielectric strength of the tape, when tested in accordance with the procedure described in Section 18, shall be not less than 350 v. per mil of thickness.

Section 8.—Renumber as "7" and change to read as follows:

> 7. The tape shall conform to the following nominal dimensions when tested in accordance with the procedure described in Section 21:

Width, in.	Thickness, in.	Length, ft.
$\frac{3}{4}$	0.030	30
$\frac{3}{4}$	0.030	15[a]
2	0.030	15

---

[a] Unless otherwise specified on the order, $\frac{3}{4}$ in. tape shall be supplied in rolls having a nominal length of 30 ft.

Section 13 (a).—Change the period of acceptance from "four" to "eight" months and the reference to ' Section 18" instead of "Section 19."

New Section.—Add the following as a new Section 21:

> 21. *Chemical Analysis.*—The chemical analysis for free sulfur shall be made in accordance with Section 16 of the Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297).

Tentative Methods of Testing Rubber Insulated Wire and Cable (**D 470 – 46 T**).[3]—The following revisions are recommended in order to make these methods more definite and workable.

Section 7 (c).—Change the 48 hr. limit for testing aged specimens to read "96 hr."

Section 14 (b).—Add the words "and vulcanized" to the end of the second sentence.

Section 27 (a).—Delete the word "of" between the words "Soluble" and "starch" in the first line.

Section 28 (b).—In the fifth sentence, add the words "of at least 25 cu. ft. per hour capacity" after the words "calibrated flow meter."

Section 30.—Change the third sentence to read: "The manometer should indicate a pressure of approximately $\frac{1}{2}$ in. of water above atmospheric in the test chamber." In the fifth sentence, change the phrase "for a few minutes" to read "for at least 15 min." In the sixth sentence, insert the words "or the rate of flow of air" after the words "ozone generator."

Tentative Methods of Testing Automotive Air Brake and Vacuum Brake Hose (**D 622 – 44 T**).[3]—The following revisions are recommended in order to bring these test methods in line with specifications for brake hose which have been adopted by the Society of Automotive Engineers:

Section 1.—Delete the last sentence since the proposed revision of vacuum brake hose will include a tension and elongation test for the cover.

Section 3 (a).—Change the length of the test sample for vacuum brake hose from "approximately 10 ft." to "approximately 15 ft."

Table I.—Add the following requirements for $\frac{5}{8}$ in. vacuum brake hose:

Inside Diameter of Hose, in.	Length of Specimen, in.	Dimensions of Form, in. (see Fig. 1)			
		A	*B*	*R*	*C* (min.)
5/8........	12	5½	4½	2¼	7/8

Section 7.—Change the second sentence to read as follows: "The assembly shall be placed for the time and temperature specified by the detailed specification in an air oven conforming to that described in the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573)."

Table II.—Add to this table the $\frac{5}{8}$ in. size of vacuum brake hose with a specified length of "22" in.

Section 13 (c).—Change to read as follows:

> (*c*) The test specimen shall be placed longitudinally in the arbor press with the fabric laps, if any, on either side and not in the line of the applied pressure. The weighing device shall be inserted in the press and the specimen placed on it so that the load applied may be measured. The test specimen shall be compressed to the form shown in Fig. 5 with dimension *D* as prescribed in Table III. The observed load required to compress the specimen to the specified dimension *D* shall be recorded. The test specimen shall then be compressed four additional consecutive times to the form shown in Fig. 5 with dimension *D* as prescribed in Table III. The specimen shall be held under load each time for 5 sec. and shall be allowed to recover for approximately 10 sec. which shall elapse between load applications. While under each application of the load, dimension *D* shall be measured by means of the proper feeler gage for the size of hose being tested. The observed load required in the fifth application to compress the specimen to the specified dimension *D* shall be recorded. The specimen shall be considered to have failed to meet the test unless this load is less than 70

lb. on the first application and unless this load exceeds 40 lb. on the fifth application.

Section 14 (a).—Change the first sentence to read as follows:

> A specimen of vacuum brake hose 12 in. in length shall be filled with di-isobutylene[a] enclosed by means of suitable cork stoppers to prevent loss by evaporation or leakage.

> [a] The di-isobutylene shall have the following characteristics:
>
> | | |
> |---|---|
> | Gravity at 60 F. | 0.718 to 0.725 |
> | Acidity | neutral to phenolphthalein |
> | Color, max. | +25 Saybolt |
> | Sulfur, per cent by weight | 0.06 |
> | Gum, max., mg. per 100 ml. | 5 |
> | Distillation min. | 212 F. |
> | Distillation max. | 221 F. |

In sentences three and four of this paragraph, change "naphtha" to read "di-isobutylene."

New Sections.—Add the following two new Sections 15 and 16, renumbering the present Section 15 as 17:

> 15. *Tensile Strength and Elongation of Cover.*—The tensile strength and elongation of the cover shall be determined in accordance with Sections 8 to 11 of the Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380), except that the test specimen shall be prepared from the original sample without having been subjected to any previous test.
>
> 16. *Adhesion Test.*—Adhesion tests between component parts of the hose shall be conducted as required on specimens prepared from the original sample without having been subjected to any previous test and the test shall be made in accordance with Sections 13 (*b*) and 14 (*a*) and (*c*) of the Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380).

Tentative Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by the Test Tube Method (**D 865 – 47 T**):[4]

Section 8 (b).—Change the 48 hr. limit for a rest period prior to physical tests to read "96 hr." This change will not materially affect the test results and will provide greater flexibility in ordinary routine control testing, particularly over week-ends.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in five standards, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Insulated Wire and Cable: Performance Rubber Compound (**D 353 – 41**):[3]

Table III.—Change this table on Insulation Resistance and the insulation constant "K" to conform to the value of 10,560 instead of 21,120, identical with Table III in Specifications D 469 – 46.

Standard Methods of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen Pressure Method (**D 572 – 42**)[3] *and by the Oven Method* (**D 573 – 45**),[3] and the *Standard Method of Air Pressure Heat Test of Vulcanized Rubber* (**D 454 – 41**).[3]—Change the 48 hr. limit for rest periods after aging and before determination of the physical properties to read "96 hr." in the appropriate sections of these three methods. The reason for this revision is the same as mentioned above for the revision of Tentative Method D 865.

Standard Method of Test for Tear Resistance of Vulcanized Rubber (**D 624 – 44**).[3]—The following revisions are recommended to provide for the use of an additional type of specimen which is known as the Graves die shape. This specimen has been shown to give more uniform results than others and is being used to an increasing extent. The committee feels that it is desirable, therefore, to include in this standard method a procedure covering its use.

Section 1.—Change line 1 to read as follows: "These methods describe the procedures for determining." Change the beginning of the second sentence to read: "They do not apply. . . " Change the beginning of the fourth sentence to read: "These methods are useful therefore only for laboratory comparisons and are not. . . . "

---

[4] 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

Designate the present Section 1 as Paragraph *(a)* and add the following as Paragraph *(b)*:

> *(b)* These methods cover procedures using three independent specimen shapes, as follows:
>
> *(1)* A razor-nicked crescent specimen,
>
> *(2)* A razor-nicked crescent specimen with tab ends, and
>
> *(3)* An unnicked 90-deg. angle specimen.
>
> No correlation between data obtained by these specimen designs is implied.

Section 5.—Change "die" to "dies" in the heading of this section.

Replace the first three sentences with the following:

> The specimens for tear resistance tests shall be stamped out with one of the steel dies conforming to the dimensions shown in Fig. 1, the cutting edges of which shall be kept sharp and free of all nicks to avoid leaving ragged edges on the specimens. It is important that the apex of the 90-deg. angle die be sharpened to provide a sharp corner. Good cutting edges can best be obtained by careful honing. Cutting shall be facilitated by lubricating the rubber surface with a lubricant of water containing 0.5 per cent of sodium chromate before dieing out each test specimen.

Change the note at the end of this section to read as follows:

> NOTE.—It is a well-known fact that vulcanized rubber exhibits a grain effect which influences its physical properties. In the case of heavily loaded compounds, the effect may produce a pronounced difference in physical properties. The grain direction shall be clearly marked on materials to be tested. Usual practice is to test with the grain running the long way of the specimen. Data so obtained shall be recorded as *tear resistance* (pulling) *with the grain*, and it is assumed that unless otherwise specified, all test specimens are to be prepared in this manner. Where grain effects are significant and are to be evaluated, an additional set of test specimens shall be cut with the grain running across the specimen. Data so obtained shall be recorded as *tear resistance* (pulling) *across the grain*.

Section 6 (a).—Change the first sentence to read as follows and delete the rest of this paragraph: "The test specimens shall conform in shape to die A, B, or C as shown in Fig. 1.

Section 6 (c).—Add "For die A and die B specimens," to the beginning of the first sentence. In the fourth sentence of this paragraph, change "with soap solution" to "with a solution as specified in Section 5."

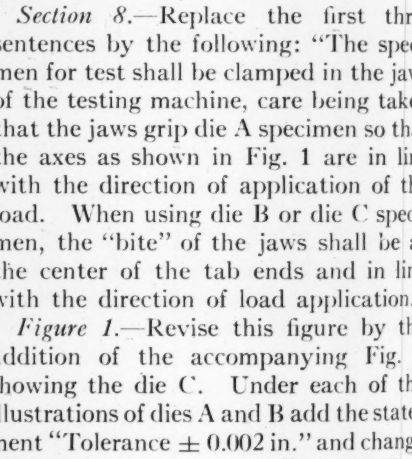
Add the following as a new Paragraph *(d)*: "*(d)* Die C specimens are not nicked."

Section 7.—Change to read as follows:

> 7. Three specimens per sample shall be tested for tear resistance. The value reported shall be the median or middle-most of those observed. If any values deviate more than 20 per cent from this median, two additional specimens shall be tested and the median of all five values shall be reported.

Section 8.—Replace the first three sentences by the following: "The specimen for test shall be clamped in the jaws of the testing machine, care being taken that the jaws grip die A specimen so that the axes as shown in Fig. 1 are in line with the direction of application of the load. When using die B or die C specimen, the "bite" of the jaws shall be at the center of the tab ends and in line with the direction of load application."

Figure 1.—Revise this figure by the addition of the accompanying Fig. 1 showing the die C. Under each of the illustrations of dies A and B add the statement "Tolerance ± 0.002 in." and change the figure caption to read: "Dies for Tear Test Specimens."



Tolerance ± 0.002"

FIG. 1.—Die C.

EDITORIAL CHANGE IN METHOD

Tentative Methods of Testing Rubber Adhesives (**D 816 - 46 T**).—The Committee recommends the following editorial change in these methods:

Section 32.—Add the following as a new Item (*3*), renumbering the subsequent items accordingly: "Description of special materials used."

PROPOSED METHOD

Proposed Tentative Method of Measuring Low Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus.[5]—The Gehman torsional apparatus for low-temperature stiffening of rubber and rubber-like materials is now commercially available, and the method is being quite widely employed in the rubber industry—particularly, in the synthetic rubber field. In the interest of standardization, it seems very desirable to have a published method available as early as possible. Agreement has not been reached on all details and further changes will undoubtedly be necessary, but the committee considers publication of the proposed method at this time very advisable.

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction other than those referred to in this report be continued as tentative. Work is under way on revision of a number of these, and the adoption as standard must be deferred until this is completed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTIES

All of the subcommittees of Committee D-11 have been exceedingly active during the year and considerable reorganization has been in progress. There have been a number of changes in subcommittee chairmen due to the retirement or resignation of the former chairmen. The loyal service of the retiring subcommittee chairmen and the ready cooperation of the new appointees is greatly appreciated by the committee. The following changes have been made. L. Cranston replaced A. M. Finley as chairman of Subcommittee I on Hose. Gordon Thompson became chairman of Subcommittee IV on Protection of Persons from Electric Shock replacing H. S. Vassar. J. T. Blake is the new chairman of Subcommittee V on Insulated Wire and Cable replacing R. A. Schatzel. R. H. Titley became chairman of Subcommittee IX on Insulating Tape replacing W. H. S. Youry. L. V. Cooper, formerly chairman of Subcommittee XVII on Hardness Tests is heading Subcommittee X on Physical Tests in place of H. M. Frecker, Jr. and is in turn replaced as chairman of Subcommittee XVII by S. R. Doner. R. H. Taylor is chairman of Subcommittee XIV on Abrasion Tests in place of G. S. Haslam and G. C. Maassen is replacing H. E. Outcault in the chairmanship of Subcommittee XV on Life Tests. B. S. Garvey, Jr. has again become active in Committee D-11 and is the new chairman of Subcommittee XVIII on Flexing Tests in place of Walter Geldard. L. E. Cheyney has replaced A. H. Flower as chairman of Subcommittee XX on Adhesion Tests.

Two new subcommittees have been organized. These are Subcommittee III on Tests of Thread Rubber and Subcommittee VII on Rubber Latices. The Thread Rubber Subcommittee is headed by J. J. Allen as chairman and has for its assignment the development of test methods for rubber thread used in elastic garments, golf balls, and similar applications. Heretofore, there has been very little, if any, standardization work in this field and because of its fairly large volume and the relatively high monetary value of the products made from it, standardization is believed to be very important. The subcommittee held its first meeting at Washington, D. C., in March, 1948, and has already organized an extensive program for development of the more important standards.

---

[5] See p. 425.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

The other new subcommittee, that on Rubber Latices of which G. H. Barnes is chairman held its organization meeting in December, 1948, and has met several times since. Here again, standards are badly needed as rubber latices return to the market because of their very extensive use in cellular rubbers, dipped and coated products, and many other applications. The subcommittee has in preparation proposed specifications for natural rubber latices.

These specifications are needed because of the large number of new producers, particularly natives in the Far East, who are entering the field since the war and are likely to put on the market latices of doubtful quality. Because of the limited acceptance of A.S.T.M. in the field of crude rubber production and due to the fact that establishment of crude rubber or latex specifications will have a definite impact on present rubber markets, it seems desirable to have the actual specifications promulgated first by agencies already active in crude rubber markets such as the Rubber Manufacturers Association or others already in the field. It is contemplated therefore that the specifications when completed during the coming year will be published in the form of proposed recommendations as a matter of information and in this form, will be referred to these other bodies for consideration.

A special committee has been organized under the chairmanship of A. E. Juve for the development of a project in cooperation with the National Bureau of Standards looking toward the establishment of a source of supply of standard reference compounds of guaranteed quality which can be used for calibration purposes in the various rubber laboratories as well as to provide reference test specimens of known quality against which other results can be compared. This committee has already made considerable progress and it is hoped that this service which has long been needed in rubber testing can be arranged in the near future.

A great deal of progress is being made in a wide variety of projects in nearly all of the established subcommittees. Detailed consideration and report of this progress is beyond the scope of this report but the results will be presented as they become available.

This report has been submitted to letter ballot of the committee, which consists of 181 members; 134 members returned their ballots, of whom 122 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

APPENDIX

PROPOSED METHOD OF MEASURING LOW-TEMPERATURE STIFFENING OF RUBBER AND RUBBER-LIKE MATERIALS BY THE GEHMAN TORSIONAL APPARATUS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes the use of the Gehman torsional apparatus for measuring the relative stiffening of rubber and rubber-like materials at low temperatures.

Apparatus

2. The apparatus[2] shall consist of the following (see Figs. 1 and 2):

(*a*) *Cylindrical Rack, C.*—The cylindrical rack in which the test specimens are held shall be composed of top and bottom disks, *D*, *D'*, made of Micarta to which are attached Micarta grips *B*, *B'*, for holding the test specimens. The hollow centerpost, *E*, and the external freely turning studs, *F*, shall be made of brass or stainless steel.

(*b*) *Insulated Chamber, G.*—The insulated chamber shall be made of an inner aluminum tube 6.5 in. in length and 3 in. in inside diameter and an outer concentric tube of chromium-plated steel, 4.5 in. in outside diameter. The space between the tubes shall be filled with an insulating material, such as Fiberglas, held in place by Micarta rings pressed on the ends of the tubes. A Micarta disk having a center hole with a diameter of 0.375 in. shall be pressed in the bottom of the inner tube. The temperature in any portion of the chamber shall be uniform within plus or minus 1 C.

(*c*) *Torsion Apparatus.*—Torsion apparatus for measuring the stiffening of the test specimens shall consist of a torsion head, *H*, and a torsion wire, *I*, of tempered hard-drawn spring wire 0.012 in. in diameter and 2.25 in. in length. The torsion wire shall be protected by a loosely fitting sleeve, *J*. A pointer, *K*, and a movable protractor, *L*, graduated in degrees, shall be provided to permit convenient and exact adjustment of the zero point.

(*d*) *Stand, M.*—A stand of stainless steel for supporting the torsion apparatus and the insulated chamber.

(*e*) *Container, N.*—An aluminum container containing a flat spiral of copper tubing, *P*, for precooling a stream of air introduced through the hole of 0.375-in. diameter in the bottom of the insulated chamber, *G*.

(*f*) *Jacket, O.*—A wooden jacket in which the aluminum container, *N*, is set.

(*g*) *Electrical Heater.*—A small cylindrical 75-w. electrical heater for insertion in the center of post, *E*, when it is desired to raise the temperature in the insulated chamber more rapidly than the ordinary heat flow into the chamber permits.

(*h*) *Thermocouple, Q, and Millivoltmeter.*—A thermocouple for measuring the temperature in the insulated chamber shall have one junction connected to the millivoltmeter and the other junction immersed in ice and water in a thermos bottle. Suitable resistance shall be inserted in the circuit so that the millivoltmeter reads directly in degrees Centrigrade.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials. Published as information, June, 1948.

[2] The apparatus was described and typical examples of the results of its use were given in a paper by Gehman, Woodford and Wilkinson, *Ind. and Engr. Chemistry*, Vol. 39, September, 1947, p. 1108.

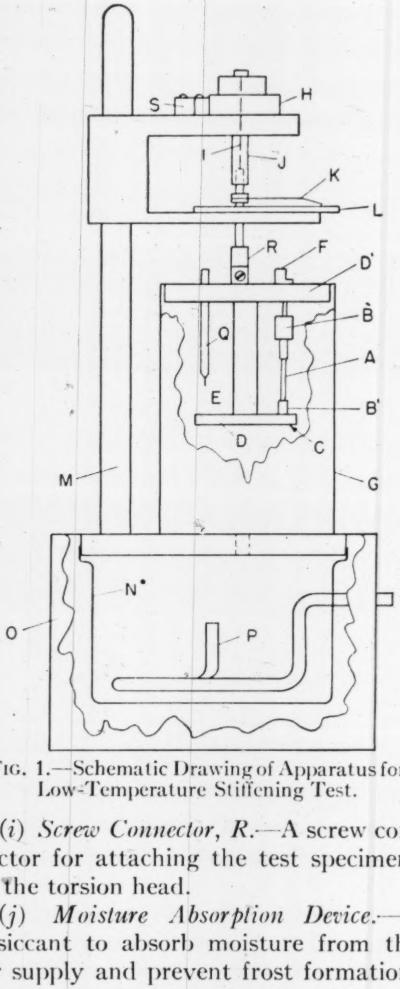


FIG. 1.—Schematic Drawing of Apparatus for Low-Temperature Stiffening Test.

(*i*) *Screw Connector, R.*—A screw connector for attaching the test specimens to the torsion head.

(*j*) *Moisture Absorption Device.*—A desiccant to absorb moisture from the air supply and prevent frost formation. Passing the air over acetone in a bottle is suitable.

(*k*) *Air Supply.*—An air supply capable of manual adjustment for rate of flow.

(*l*) *Microswitch, S.*—A microswitch mounted on the stand, *M*, actuated by a cam on the torsion head at the end of a 180-deg. rotation.

(*m*) *Electric Timer.*—An electric timer connected to the microswitch, *S*. The timer shall give an audible indication at the end of a 10-sec. period after being turned on by the microswitch, *S*.

Test Specimens

3. The test specimens shall be cut with a suitable die from tensile test sheets. Small rubber strips 1.625 in. in length, 0.125 in. in width and 0.079 in. in thickness are suitable for this test. Since the test method obtains relative results, the width and gage of the specimens are not critical. It is recommended that gage variations do not exceed those normally encountered in molded test sheets.

Procedure

4. (*a*) The test specimens shall be mounted in the rack. The rack shall be set in the insulated chamber and the chamber, in turn, shall be placed on the stand.

(*b*) One of the specimens shall be connected to the torsion head by means of the screw connector. The pointer reading shall be adjusted to zero by rotating the protractor scale. The torsion head shall be turned 180 deg. and the pointer reading, after a period of 10 sec. as indicated by the electric timer, shall be recorded. (If the reading does not fall in the range of 170 to 120 deg. the specimens are not suitable for testing with the standard torsion wire and a lighter or heavier wire should be used to bring the reading into the acceptable range of 170 to 120 deg.)

(*c*) The torsion head shall be returned to its initial position and the specimen disconnected. The insulated chamber shall then be rotated to bring the next specimen into position for measurement and the procedure of Paragraph (*b*) shall be repeated. All the specimens in the rack shall be measured in this manner. The room temperature at which these measurements are made shall be recorded.

(*d*) The rack shall be removed from the chamber and dry ice shall be introduced into the chamber to a depth of about 2 in. (The chamber may be packed full of dry ice temporily to cool the walls more rapidly.) The rack shall then be replaced in the chamber. The aluminum container shall be filled with dry ice and the dry ice shall be moistened with acetone. The stand shall be set on the aluminum container and the insulated chamber shall be set on the stand. The thermocouple shall be inserted in the chamber and the air flow shall be started and continued until the temperature in the chamber has dropped to the lowest test temperature desired. (This varies with the type of material being tested since time is saved by not starting at a temperature more than 10 C. lower than the freezing point of the material; for natural rubber stocks, the lowest temperature required is usually −70 C.) When this temperature is reached the air flow shall be reduced and regulated to maintain a constant temperature for 5 min. The measurement of the specimens as was done at room temperature shall be repeated.

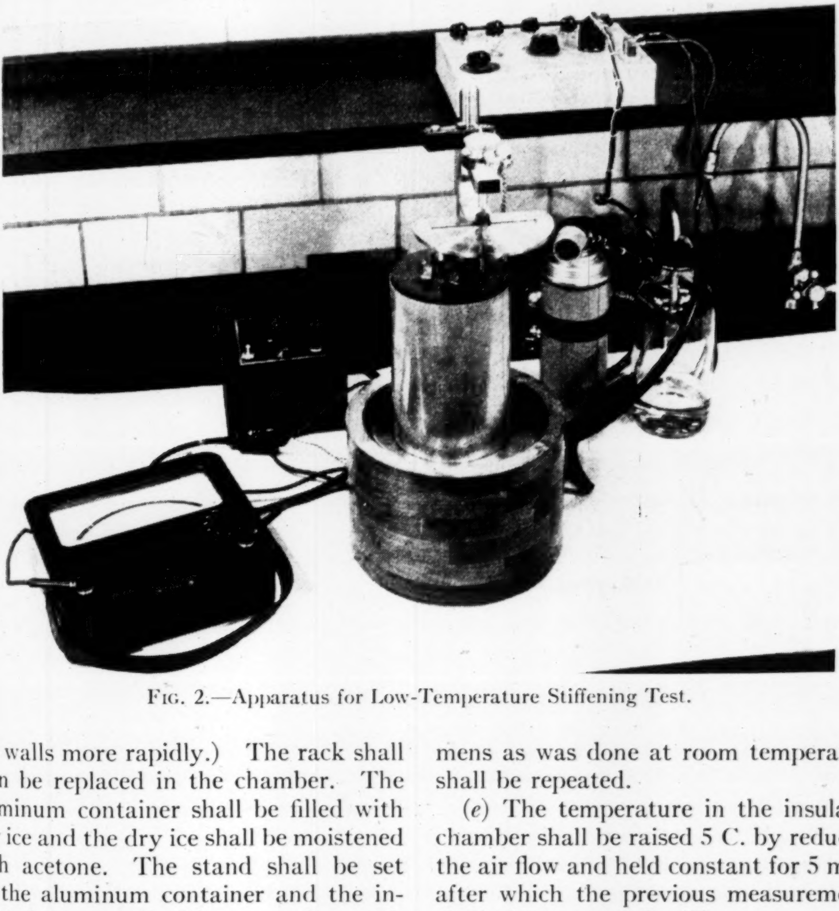


FIG. 2.—Apparatus for Low-Temperature Stiffening Test.

(*e*) The temperature in the insulated chamber shall be raised 5 C. by reducing the air flow and held constant for 5 min., after which the previous measurements shall be repeated. The temperature shall be subsequently raised in 5-C. steps and the specimens measured at each temperature until the pointer reading is within 5 to 10 deg. of the original room temperature reading.

(*f*) Steps of 10 C., instead of 5 C., may be used if desired, for the less sensitive parts of the curve. The temperature rise may be accelerated by use of the auxiliary heater described in Section 2 (*g*). The test may be shortened by concluding the temperature rise as soon as the range of interest has been passed as described in Section 5 (*c*).

(*g*) *Long-Term Cold Hardening.*—For long-term cold hardening tests at a given temperature additional racks are required. The test specimens shall be mounted in the racks and the pointer deflection at room temperature recorded for each specimen. The temperature of the insulated chamber shall then be lowered to the test temperature, held for 5 min. and pointer readings recorded for each specimen. The racks of specimens shall be stored in a cold box or cold room whose temperature is regulated at the desired value and the deflections shall be measured periodically. In carrying out these measurements the insulated chamber shall be introduced into the cold box long enough for its temperature to be reduced to the test temperature. A rack of specimens then shall be placed in the chamber and the apparatus transferred from the cold box to the measuring stand. The temperature in the insulated chamber shall be maintained at the test temperature by air flow through dry ice in the aluminum container in the usual way. After the specimens are measured, they shall be returned to the cold box in the insulated chamber, the rack holding them removed from the chamber and a new rack inserted in the chamber and taken to the stand for measurement.

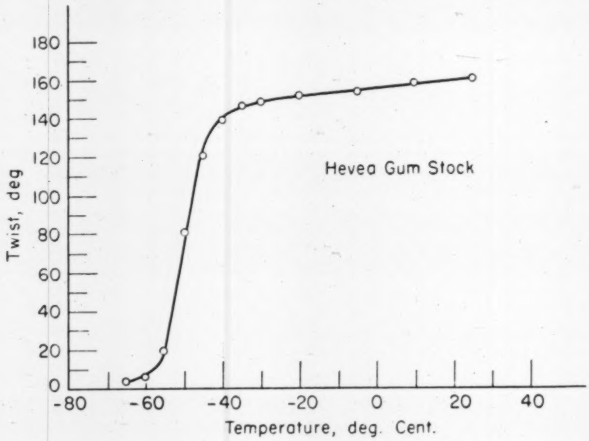


FIG. 3.—Illustrative Chart of Twist *versus* Temperature.

Calculations

5. (*a*) A plot shall be made of the pointer reading, or angle of twist of the test specimen, against the temperature, as illustrated in Fig. 3. The torsional modulus of the specimen at any temperature is proportional to the quantity $\frac{180\text{ deg.} - \text{twist}}{\text{twist}}$. Table I lists the value of this factor for every degree from 1 to 180.

(*b*) The relative modulus at any temperature is the ratio of the modulus at that temperature to the modulus at 25 C. The value of the relative modulus for any temperature is readily determined from the angles of twist at that temperature and at 25 C., as given by the twist *versus* temperature plot (see

Fig. 3), and the ratio of the factors $\frac{180\text{ deg.} - \text{twist}}{\text{twist}}$ corresponding to those angles from Table I. The relative modulus is the ratio by which the stiffness has increased over its value at room temperature.

TABLE I.—MODULUS PROPORTIONALITY FACTORS.

Twist, X, deg.	$\frac{180 - X}{X}$	Twist, X, deg.	$\frac{180 - X}{X}$	Twist, X, deg.	$\frac{180 - X}{X}$	Twist, X, deg.	$\frac{180 - X}{X}$
1	179	51	2.53	101	0.782	151	0.192
2	89	52	2.46	102	0.765	152	0.184
3	59	53	2.40	103	0.748	153	0.176
4	44	54	2.33	104	0.731	154	0.169
5	35	55	2.27	105	0.714	155	0.161
6	29	56	2.21	106	0.698	156	0.154
7	24.7	57	2.16	107	0.682	157	0.146
8	21.5	58	2.10	108	0.667	158	0.141
9	19.0	59	2.05	109	0.651	159	0.132
10	17.0	60	2.00	110	0.636	160	0.125
11	15.4	61	1.95	111	0.622	161	0.1180
12	14.0	62	1.90	112	0.607	162	0.1111
13	12.8	63	1.86	113	0.593	163	0.1043
14	11.86	64	1.81	114	0.579	164	0.0975
15	11.00	65	1.77	115	0.565	165	0.0909
16	10.25	66	1.73	116	0.552	166	0.0843
17	9.59	67	1.69	117	0.538	167	0.0778
18	9.00	68	1.65	118	0.525	168	0.0714
19	8.47	69	1.61	119	0.513	169	0.0651
20	8.00	70	1.571	120	0.500	170	0.0588
21	7.57	71	1.535	121	0.488	171	0.0527
22	7.18	72	1.500	122	0.475	172	0.0465
23	6.83	73	1.466	123	0.463	173	0.0405
24	6.50	74	1.432	124	0.452	174	0.0345
25	6.20	75	1.400	125	0.440	175	0.0286
26	5.92	76	1.368	126	0.429	176	0.0227
27	5.67	77	1.337	127	0.417	177	0.0169
28	5.43	78	1.308	128	0.406	178	0.0112
29	5.21	79	1.278	129	0.395	179	0.0056
30	5.00	80	1.250	130	0.385	180	0
31	4.81	81	1.222	131	0.374		
32	4.62	82	1.195	132	0.364		
33	4.45	83	1.169	133	0.353		
34	4.29	84	1.143	134	0.343		
35	4.14	85	1.118	135	0.333		
36	4.00	86	1.093	136	0.324		
37	3.86	87	1.069	137	0.314		
38	3.74	88	1.045	138	0.304		
39	3.62	89	1.022	139	0.295		
40	3.50	90	1.000	140	0.286		
41	3.39	91	0.978	141	0.277		
42	3.29	92	0.956	142	0.267		
43	3.19	93	0.935	143	0.258		
44	3.09	94	0.915	144	0.250		
45	3.00	95	0.895	145	0.241		
46	2.91	96	0.875	146	0.233		
47	2.83	97	0.856	147	0.224		
48	2.75	98	0.837	148	0.216		
49	2.67	99	0.818	149	0.208		
50	2.60	100	0.800	150	0.200		

(*c*) To determine the temperatures at which the relative modulus is 2, 5, 10 and 100, Table II shall be used in conjunction with the twist *versus* temperature curve for the specimen. The first column of Table II lists each degree in the range of 120 to 170, so that the value corresponding to the twist of the specimen at 25 C. can be selected. Successive columns give the twist angles which correspond to values of 2, 5, 10 and 100 for the

TABLE II.—TWIST ANGLES FOR DESIGNATED VALUES OF THE RELATIVE MODULUS.

Twist at 25 C., deg.	Twist for R. M. = 2, deg.	Twist for R. M. = 5, deg.	Twist for R. M. = 10, deg.	Twist for R. M. = 100, deg.
120	90	51	30	3
121	91	52	31	4
122	92	53	31	4
123	93	54	32	4
124	95	55	33	4
125	96	56	33	4
126	97	57	34	4
127	98	58	35	4
128	99	59	36	4
129	101	61	36	5
130	102	62	37	5
131	103	63	38	5
132	104	64	39	5
133	105	65	40	5
134	107	66	41	5
135	108	68	42	5
136	109	69	42	5
137	111	70	43	6
138	112	71	45	6
139	113	72	46	6
140	114	74	47	6
141	116	75	48	6
142	117	77	49	7
143	119	78	50	7
144	120	80	51	7
145	121	82	53	7
146	123	83	54	7
147	124	85	55	7
148	124	85	56	8
149	127	88	58	8
150	129	90	60	9
151	130	92	62	9
152	132	94	62	9
153	133	96	65	10
154	134	97	67	10
155	136	100	69	11
156	138	102	71	11
157	139	104	73	12
158	140	106	75	12
159	142	108	78	13
160	144	111	80	13
161	146	113	82	14
162	147	116	85	15
163	149	118	88	16
164	151	121	91	17
165	152	124	94	18
166	154	126	98	19
167	156	130	101	20
168	158	133	105	22
169	159	136	109	24
170	161	139	113	26

relative modulus. The temperatures corresponding to these angles are then read from the twist *versus* temperature curve for the specimen and are designated as T_2, T_5, T_{10} and T_{100} respectively. Table II can be used during a test to determine when a particular T value has been obtained so that the test may then be concluded.

NOTE: *Example.*—The twist *versus* temperature curve for a Hevea gum stock is given in Fig. 3. From this curve the twist at 25 C. is found to be 160 deg. Referring to Table II the angles of twist corresponding to relative modulus values of 2, 5, 10 and 100 are respectively 144, 111, 80 and 13. Referring again to the curve in Fig. 3 the temperatures at which these angles of twist occur are found to be −38 C., −47 C., −50 C. and −56 C., respectively.

Report

6. (*a*) The report shall include the temperatures at which the relative modulus is 2, 5, 10 and 100. These temperatures shall be designated respectively as T_2, T_5, T_{10} and T_{100}. It shall also include the 300 per cent stress-strain modulus of the material at room temperature to be used as a basis for judging the stiffness of the specimens as well as the effect of temperature on stiffness.

(*b*) *Long-Term Cold Hardening.*—For long-term cold hardening tests, the results shall be presented as plots of the ratio of modulus to original modulus at the test temperature *versus* time, the modulus ratio being plotted on a logarithmic scale, as illustrated in Fig. 4.

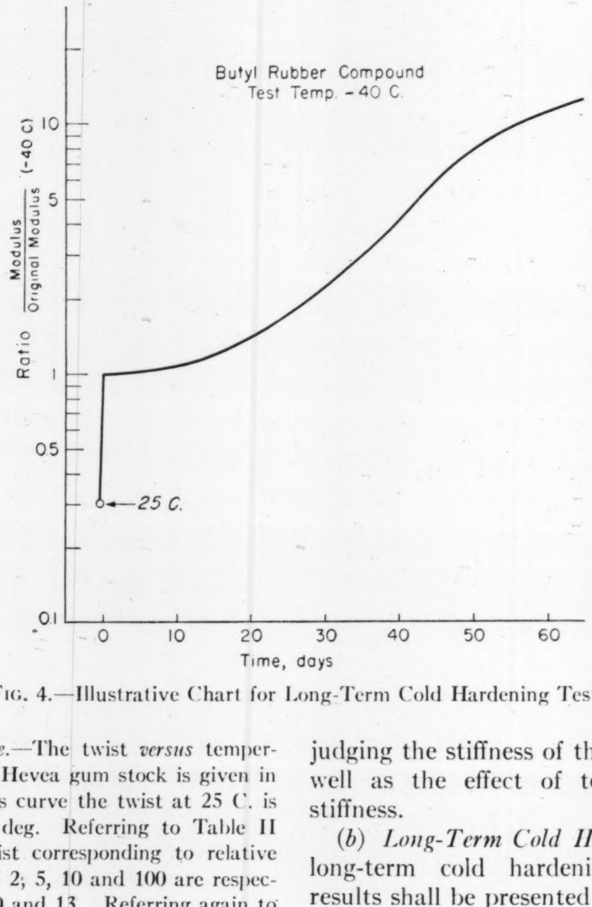


FIG. 4.—Illustrative Chart for Long-Term Cold Hardening Test.

REPORT OF COMMITTEE D-12*

ON

SOAPS AND OTHER DETERGENTS

The annual meeting of Committee D-12 on Soaps and Other Detergents was held in New York, N. Y., on March 23 and 24, 1948.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, F. W. Smither.
Vice-Chairman, F. Krassner.
Secretary, J. C. Harris.
Advisory Committee: F. W. Smither, G. P. Fulton, M. L. Sheely, W. G. Morse, W. H. Koch, L. B. Parsons, F. Krassner, J. C. Harris, J. B. Crowe.

NEW TENTATIVE

The committee recommends for publication the Proposed Tentative Method for Sampling and Chemical Analysis of Borax, as appended hereto.[1] This method, when adopted, is to be added to the Standard Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 46).

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 47 T)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[3] of the following three standards be adopted as standard and incorporated in the specifications:

Standard Specifications for:

Compound Chip Soap (with Rosin) (D 690 – 44),
Milled Toilet Soap (D 455 – 39), and
White Floating Toilet Soap (D 499 – 39).

EDITORIAL CHANGE IN STANDARD

The committee recommends the following editorial change in the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 46):[3]

Section 37.—Change the second sentence to read as follows by the addition of the italicized words: "In a 250-ml. Erlenmeyer flask dissolve 2 g. of the fatty acids, accurately weighed, in 20 to 30 ml. of neutral 95 per cent ethyl alcohol *near the boiling point.*"

Section 41 (c).—Change the factor for calculating borax from "0.00955" to read "0.009536."

Section 42 (d).—Change the factor for calculating the weight of SiO_2 to sodium silicate from "1.308" to read "1.318."

The committee also recommends that the title of Standard Method D 501 – 46 be changed editorially from "Methods of Sampling and Chemical Analysis of Special Detergents" to "Methods of Sampling and Chemical Analysis of Alkaline Detergents." The revised title designates more clearly what is meant and should prove less confusing.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] This method was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[2] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[3] 1946 Book of A.S.T.M. Standards, Part III-A, pp. 1253 and 1254.

REAFFIRMATION OF STANDARDS

The committee recommends reaffirmation of the following 12 standards which have been published for six years or longer without revision and which are to be continued as such as no revisions are recommended at this time:

Standard Specifications for:[4]

Chip Soap (D 496 – 39),
Ordinary Bar Soap (D 497 – 39),
Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39),
Alkaline Soap Powder (D 534 – 42),
Palm Oil Solid Soap (Type A, Straight; Type B, Blended) (D 535 – 41),
Palm Oil Chip Soap (Type A, Straight; Type B, Blended) (D 536 – 42),
Sodium Metasilicate (D 537 – 41),
Olive Oil Solid Soap (Type A, Straight; Type B, Blended) (D 592 – 42),
Salt-Water Soap (D 593 – 42),
Sodium Sesquisilicate (D 594 – 41), and
Olive Oil Chip Soap (Type A, Straight; Type B, Blended) (D 630 – 42).

Standard Method of Test for:

Particle Size of Soaps and Other Detergents (D 502 – 39).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-1 on Soaps (L. K. Whyte, chairman) recommended the editorial change in Standard Methods D 460 referred to earlier in this report.

Subcommittee T-3 on Dry Cleaning (G. P. Fulton, chairman).—Cooperative work on moisture determination of dry cleaning detergents is expected to be accomplished in the coming year. The committee expects to complete preparation of a performance specification within the next year.

Subcommittee T-4 on Special Detergents (W. H. Koch, chairman) prepared the new Tentative Method for Sampling and Chemical Analysis of Borax.[1] Revision of the method for analysis of sodium bicarbonate will be undertaken to provide both a rapid, moderately accurate method, as well as an alternative precise one. Further plans were deferred until a chairman for Subcommittee S-4 has been appointed.

Subcommittee T-5 on Physical Testing (J. A. Woodhead, chairman).—Cooperative examination of the canvas disk method for determination of wetting evaluation of surface active agents indicated that closer control of certain test factors must be made before it could be recommended for publication as tentative. Investigation of the skein wetting test will also be accomplished during the coming year. Chairmen of task committees will be named for development of other tests such as lathering and detergency, so that other recommendations may be presented to the committee.

Subcommittee T-6 on Metal Cleaning (J. C. Harris, chairman).—Certain revisions of the Tentative Method of Total Immersion Corrosion Test of Water-Soluble Aluminum Cleaners (D 930 – 47 T) are to be made during the year by the subcommittee, to be presented for action at the next annual meeting of the committee. Proposed methods for corrosion of metals other than aluminum have been developed, as well as methods for determination of buffer index and surface and interfacial tension. Critical examination will be made of the Tentative Method for

---

[4] 1946 Book of A.S.T.M. Standards, Part III-A

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T) as applied to metal cleaning. Editorial changes in Section 34 of Standard Methods of Chemical Analysis of Industrial Metal Cleaning Compositions (D 800 - 45) will be made to require the use of A.S.T.M. Specifications for Nitration Grades of Toluene and Xylene (D 841 and D 843) for the moisture determination by the distillation method.

Subcommittee S-1 on Soaps (F. Krassner, chairman).—Two specifications to cover solid, and granular or chipped low and medium-titre soap have been proposed but require further consideration by the subcommittee before recommendation for approval.

Subcommittee G-2 on Nomenclature and Definitions (C. A. Marlies, chairman).—Recommended that Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 47 T) be adopted as standard. Further action by this subcommittee will comprise cooperative effort with the individual subcommittees requiring definitions. This has the advantage of utilizing the abilities of the various members of the specific subcommittees without need for separate meetings.

This report has been submitted to letter ballot of the committee, which consists of 96 members; 86 members returned their ballots, of whom 84 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. S. VAN ZILE,
Chairman.

J. C. HARRIS,
Secretary.

REPORT OF COMMITTEE D-13[*]
ON
TEXTILE MATERIALS

Committee D-13 on Textile Materials held two 3-day meetings during the year. The fall meeting was in Philadelphia, Pa. on October 15 to 17, 1947, with a registered attendance of 261 members and guests; the spring meeting was in New York, N. Y., on March 17 to 19, 1948, with a total attendance of 263. Each of these attendance records exceeded those of any previous meeting. Nineteen subcommittees or sections held sessions at the fall meeting and 24 at the spring meeting. Administrative guidance of the committee required two meetings of the Advisory Committee and one of the Officers Committee.

The Papers Session of the fall meeting, planned in cooperation with the Philadelphia District Council, was divided into two parts. The afternoon session featured a Symposium on Flammability at which the following papers[1] were presented: "Flammability of Textiles," by Frederic Bonnet; "Flameproofing of Textiles in Theory and Practice," by R. W. Little; and "The Retailers Viewpoint on the Flammability of Textiles," by C. W. Dorn. The second group of papers was delivered at an evening session, following the usual banquet, and the speakers and their subjects were as follows: "Textiles for Peace and War,"[2] by S. J. Kennedy; "Cotton Textile Fibers in Various Industrial Uses," by B. L. Whittier; "Modifications of Textile Fiber Properties through Finishing Operations,"[2] by D. H. Powers; and "Some of the Newer Fibers, Including Glass, in the Textile Industry,"[3] by Rene Bouvet.

The Papers Session at the spring meeting was dedicated appropriately to the memory of Dr. Harold DeWitt Smith and was featured by the following papers: "The Importance of the Time Factor in the Study of Textile Properties,"[4] by Edward R. Schwarz; "The Stress-Strain Diagram as a Tool in Textile Research,"[4] by Walter J. Hamburger; "Some Applications of Stress-Strain Measurements to the Determination of Fiber Properties,"[4] by Arnold M. Sookne.

The organization of Committee D-13 was further altered during the year in order to keep in step with changing conditions and to improve the efficiency of operation. Subcommittee A-12 on Hosiery became an actuality, under the chairmanship of E. N. Ditton and with the cooperation of the National Association of Hosiery Manufacturers. A new Subcommittee A-14 on Bonded Fabrics was authorized by the Advisory Committee, and has been organized under the joint chairmanship of Gordon Harvey, representing the A.S.T.M., and K. H. Barnard for the American Association of Textile Chemists and Colorists. In order to handle more effectively the increasing volume of work on general test methods, physical and chemical, Subcommittee B-1, Sections I on Methods of Testing and II on Instruments and Machines, and Subcommittee B-4 on

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] ASTM BULLETIN, Vol. 150, January, 1948, pp. 29–42 (TP 1–14).

[2] Abstracts of these papers were published in the ASTM BULLETIN, Vol. 149, December, 1947, pp. 33–36.

[3] An abstract of this paper was published in the ASTM BULLETIN, Vol. 150, January, 1948, p. 20.

[4] "A.S.T.M. Standards on Textile Materials," October 1948; issued as a separate publication.

Bleaching, Dyeing and Finishing were merged into a single Subcommittee B-1 on Test Methods. The supervision of this subcommittee was placed under the chairmanship of W. D. Appel, who, together with group chairmen Lewis Larrick, W. M. Scott and M. L. Staples, will act as a steering committee to organize and guide the many and important task groups required. Subcommittee B-1, Section III on Atmospheric Conditions and Regain was set up as a separate Subcommittee B-3 with the same title and the same chairman, R. H. Brown.

The following changes in subcommittee chairmanships occurred during the year: R. L. Lee, Jr., vice M. E. Campbell, as chairman of Subcommittee A-1, Section I on Cotton; F. A. Mennerich, vice F. S. Mapes, as chairman of Subcommittee A-8 on Glass Fiber and Its Products; W. H. Lehmberg, vice G. H. Harnden as chairman of Subcommittee A-10 on Felt; G. H. Hopkins, vice D. C. Scott, as chairman of Subcommittee C-3 on Membership. R. F. Tener was elected to fill the vacant chairmanship of Subcommittee A-8, Section V on Abrasion, and R. F. Caroselli to fill that of Subcommittee A-8, Section VI on Sizing.

During the year a cooperative undertaking was inaugurated between the A.S.T.M., the National Bureau of Standards, and wool blanket manufacturers for the purpose of solving certain problems involved in the determination of the wool content of individual blankets and of the degree of uniformity which may reasonably be expected from blanket to blanket as delivered into commercial channels. The Advisory Committee approved a request that Committee D-13 should take charge of the work for the Society, and leadership was assigned to and accepted by O. P. Beckwith, as chairman of Subcommittee B-5 on Sampling, Presentation and Interpretation of Data.

This same subcommittee has prepared, and Committee D-13 has approved, a "Tentative Recommended Practice for Interlaboratory Testing." For the first time it places in the hands of subcommittee chairmen a completely detailed and illustrated description of how to proceed in this important phase of their work to secure the greatest efficiency from the interlaboratory tests. It embodies the coupling of experiment planning with the techniques of analysis of variance so that the resultant data may be made to yield the maximum of reliable information.

The members of Committee D-13 were advised of a proposal under consideration by the Federal Specification Board which, if adopted, would establish new standard laboratory atmospheric conditions of 50 per cent relative humidity at 73.5 F. for all procurement testing under Federal specifications, with certain exceptions. This is a matter of such important technical and commercial concern to the textile industry that Subcommittee B-3 on Atmospheric Conditions and Regains under R. H. Brown, chairman, was instructed to canvass the entire membership of Committee D-13 to ascertain the sentiment of this representative cross-section of the various branches of the industry to such a proposal. The results of the balloting and the discussion at the spring meeting revealed a very strong objection to any change in the relative humidity from the present standard of 65 per cent to the proposed 50 per cent. Less objection was registered to a change in the temperature requirement from the present standard of 70 F. to the proposed 73.5 F. The Federal Specification Board has been advised of the results of the survey.

This year will mark the approval by Committee D-13 of an important revision of the definition of rayon. The result of the change is to recognize the cellulose ester fibers as having properties of a physical and chemical character sufficiently different from those of the regenerated cellulose fibers to warrant their classification under a new generic term of "estron."

Members of Committee D-13 have been appointed to represent the A.S.T.M. in the following activities: H. J. Ball, vice J. R. Bonnar, on Sectional Committee L-14 on Textile Test Methods under the sponsorship of the American Standards Association; Laura E. Pratt, vice R. L. Fisher, on Sectional Committee L-4 on Specifications and Standards for Sheets and Sheeting; H. J. Ball and A. G. Scroggie as American delegates to the first postwar meeting of Textile Committee 38 of the International Standards Organization at Buxton, England, June 7 to 12, 1948.

The record of the year's events must not fail to record the saddening news of the death of Alfred E. Suter on October 23, 1947. He became a member of Committee D-13 in 1916, two years after its organization, and his generous contribution of time and effort to advance its standardization activities was continually helpful over the years. He will be missed from the meetings and his characteristic qualities of modesty of manner, honesty of dealing, sincerity of purpose, and desire for scientific accuracy will remain indelibly impressed in the memory of all who knew him.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. J. Ball.
First Vice-Chairman, G. E. Hopkins.
Second Vice-Chairman, Frederic Bonnet.
Secretary, W. H. Whitcomb.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Mechanical Roll Felt (D 944 - 47 T).

Revision of Tentative:

Definitions of Terms Relating to Textile Materials (D 123 - 46 T),
General Methods of Testing Cotton Fibers (D 414 - 40 T), and
Method of Test for Colorfastness of Dyed Cellulose Acetate Rayon to Atmospheric Fumes (D 682 - 42 T).

Revision and Reversion to Tentative:

General Methods of Testing and Tolerances for Cotton Yarns (D 180 - 44),
Method of Test for Determination of Small Amounts of Copper, Manganese, and Nickel in Textiles (D 377 - 37),
Methods of Test for Fineness of Wool (D 419 - 44), and
Spec. and Methods of Test for Fineness of Wool Tops (D 472 - 41).

These recommendations were accepted by the Standards Committee on October 31, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A, and also in the 1947 Compilation of A.S.T.M. Standards on Textile Materials.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication a new tentative recommended practice, and is recommending the revision of five tentatives, tentative revisions of two standards, adoption as standard of several definitions and one tentative method as revised, and the revision of four standards and reversion to tentative. The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

All tentatives not specifically referred to are being actively studied by the respective sponsoring subcommittees.

ACTIVITIES OF SUBCOMMITTEES

All subcommittees have been active during the year. Besides the activities indicated by the recommendations appearing in the Appendix, the following should be mentioned:

Subcommittee A-1 on Cotton and Its Products (B. L. Whittier, chairman):

Section I on Cotton (R. L. Lee, Jr., chairman) is interested in having developed check samples of cotton for fiber analyses, in the measurement of fiber strength, and in the use of the Fibrograph for measuring fiber length. At a meeting of this section J. K. Phillips, Goodyear Tire and Rubber Co., discussed "The Comparison of the Pressley and Scott Machines when Used to Test Cotton Fiber Strength Using the Pressley Jaws," and "Zero *versus* Greater than Zero Length Test Specimens in both Bundle and Single Fiber Tests on the Scott IP-4 Machine."

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) is studying revisions of the Tests and Tolerances for Cotton Yarns (D 180 – 47 T).

Section VI on Narrow Cotton Fabrics (F. S. Mapes, chairman) is conducting interlaboratory tests to determine the most suitable means of measuring thickness of tapes.

Subcommittee A-2 on Man-Made Organic Base Fibers and Their Products (A. M. Tenney, chairman):

Section I on Filaments, Monofilaments, and Filament Yarns (J. B. Goldberg, chairman) is studying yarn specifications and tolerances in connection with manufacturers and yarn buyers; methods of determining friction, luster, and ash content; application of initial tension in strength tests; standards for fibers other than rayon.

Section II on Fabrics (G. H. Hotte, chairman) is studying revision of the Methods for Wet Strength of Rayon Fabrics (D 415 – 38) and for Yarn Slippage (D 434 – 42), and is developing a method of measuring "shift" in man-made fabrics.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) is continuing its studies on sampling of raw wool, raw wool shrinkage, moisture determination, yield determination of tops, noils, and waste, fineness of wool and tops, conversion factor for wool (top-noil-waste relationship). A paper on "The Determination of Crimp" by Dorothea Abbot and L. W. Rainard, Alexander Smith & Sons Carpet Co., was presented at a meeting of this section.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman) is considering the British proposal to define woolen yarn as "condenser spun" yarn.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman) is studying the determination of magnetic iron content of asbestos fiber, tolerances and methods of test for asbestos laps, and specifications for asbestos roving and lap.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (S. J. Hayes, chairman) is developing test methods for twine.

Subcommittee A-6 on Household and Garment Fabrics (Laura E. Pratt, chairman) is considering the withdrawal of the Specifications for Cotton Corduroy (D 625 – 41 T) and is studying revision of the Specifications for Bleached Cotton

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Broadcloth (D 504 - 41 T), Household Blanketing (D 576 - 40 T), Terry Toweling (D 505 - 40 T) and the discontinuance of the work on work garment fabrics.

A survey is being made to determine the need for standards on Upholstery Tapestries (D 678 - 42 T). The determination of mercerization is also being studied.

Subcommittee A-8 on Glass Fiber and Its Products (F. A. Mennerich, chairman) is studying the corrosive effects of glass textiles used in electrical insulation upon copper conductors; the effect of type of jaws on the breaking strength of glass yarn.

Subcommittee A-9 on Tire Cord and Fabrics (F. C. Kennedy, chairman) is studying the time limits in oven-dry strength testing and methods of pretensioning strength test specimens. Revisions of the Tentative Methods for Cotton and Rayon Tire Cord (D 179 - 46 T and D 885 - 46 T) are also under consideration.

Subcommittee A-10 on Felt (W. H. Lehmberg, chairman) is studying the suitability of dumb-bell shaped tension specimens; stiffness and sizing in felts; specifications for sheet felts; mildew resistance; pH value of felt. Work on hardness and resiliency has been discontinued. R. R. Stevens, The Felters Co., presented a paper on "Laboratory Methods for Evaluating the Felting Power of Wools" at the March meeting of this subcommittee.

Subcommittee A-11 on Pile Fabrics (S. L. Peebles, chairman) is studying the flame resistance of carpets, determination of equilibrium moisture, mothproofness, tests for spotting, staining, and crocking, and the National Bureau of Standards wear tester.

Subcommittee A-12 on Hosiery (E. N. Ditton, chairman) is studying tests for snag resistance, hosiery service tests, stretch, and shrinkage.

Subcommittee A-14 on Bonded Fabrics (G. B. Harvey, chairman).—This new subcommittee has drafted methods of test for weight, tensile strength, and bursting strength of bonded fabrics.

Subcommittee B-1 on Methods (W. D. Appel, chairman) has under investigation visual standards for inspection of textiles; yarn number of yarns from woven fibers; Pressley strength test for fabrics; elastic fabrics; stress-strain curves; jaws and clamps; thermal transmission; calibration of inclined plane testers; water resistance; resistance to insect pests and microorganisms, and revision of Specifications for Fire-Retardant Properties of Treated Textile Fabrics (D 626 - 41 T) with respect to "length of char."

The following papers were presented before this subcommittee: "Yarn Sheet Abrasion Tests," by E. M. Hicks, du Pont de Nemours and Co.; "Service Wearing Tests of Textiles,"[6] by E. N. Ditton, Gotham Hosiery Co.; "Solution of Problem of Producing Uniform Abrasion and Its Application to the Testing of Textiles," by H. F. Schiefer, National Bureau of Standards; and "Report of the Task Group Studying the Wyzenbeek Precision Wear Test Meter,"[7] by Seaman J. Tanenhaus and Gerald Winston, Philadelphia Quartermaster Depot.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman) has a number of definitions under consideration for such terms as webbing, tape, wool, multifilament, monofilament, lisle, and moisture equilibrium for testing purposes.

Subcommittee B-3 on Atmospheric Conditions and Regain (R. H. Brown, chairman) has carried out an extensive survey of the reaction of members of Committee D-13 to changing the standard atmosphere from 65 per cent relative humidity at 70 F. to 50 per cent relative humidity at 73.5 F. as proposed by the Federal Specifications Board and endorsed by Technical Committee X of Committee E-1 on Methods of Testing. The results of the survey showed very definitely a preference for 65 per cent relative humidity but a greater willingness for acceptance of a temperature of 73.5 F.

---

[6] ASTM BULLETIN, No. 152, May, 1948, p. 93.

[7] ASTM BULLETIN, No. 154, October, 1948, p. 74 (TP232).

Subcommittee B-5 on Sampling, Presentation and Interpretation of Data (O. P. Beckwith, chairman).—At the October meeting a paper on "Precision of Laboratory Measurements of Breaking Strength of Textiles," by J. W. Hopkins, Canadian National Research Council, was presented.

This report has been submitted to letter ballot of the committee, which consists of 341 members; 194 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1946 Book of A.S.T.M. Standards, Part III-A or in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

NEW TENTATIVE

Tentative Recommended Practice for Planning Interlaboratory Testing of Textile Materials:

This recommended practice, as prepared by Subcommittee B-5, is recommended by Committee D-13 for publication as tentative as appended hereto.[1]

REVISIONS OF TENTATIVES

Tentative Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 47 T).

The committee recommends the following revisions in several definitions, two new definitions, and the inclusion of a new test for knot breaking strength, as prepared by Subcommittee A-2:

Section 2 (a).—Change the definition of rayon to read as follows:

2. (*a*) *Rayon, n.*—A generic term for manmade fibers, monofilaments and continuous filament yarns composed of regenerated cellulose with or without lesser amounts of nonfiber-forming materials.

NOTE.—Rayon as commercially manufactured is produced by three different processes. The different type rayons are known as follows:

(*a*) Viscose Rayon
(*b*) Cuprammonium Rayon
(*c*) Nitrocellulose Rayon

Section 2 (b).—Change the definition of rayon, modified, to read as follows:

(*b*) *Rayon, Modified, n.*—Rayon composed principally of regenerated cellulose, and containing amounts of non-regenerated cellulose fiber-forming material.

Section 2 (c).—Add the following new definition of "estron" as a new Paragraph (*c*), relettering the present paragraphs accordingly:

(*c*) *Estron, n.*—A generic term for man-made fibers, monofilaments and continuous filament yarns composed of one or more esters of cellulose with or without lesser amounts of nonfiber-forming materials.

NOTE.—Estron as commercially manufactured is produced at present by one process, namely, Cellulose Acetate Estron (Acetate Estron).

Section 2 (d).—Add the following as a new Paragraph (*d*): "(*d*) *Rayon Monofilament.*—A single rayon filament."

Reletter Paragraph (*c*) as (*e*) and change to read as follows: "(*e*) *Rayon Yarns.*—An assemblage of more than one continuous rayon filament."

Reletter Paragraph (*d*) as (*f*) and change to read as follows:

(*f*) *Yarn Number (Linear Density).*—The yarn number of a rayon yarn shall be expressed in denier or grex units. Yarn number is calculated on a commercial regain basis.

---

[1] This recommended practice was accepted as tentative by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

NOTE.—Linear density expressed in the denier system equals the number of unit weights of 0.05 g. per 450 m. of yarn length. This is numerically equal to the weight in grams of 9000 m. of yarn. Linear density expressed in the grex system equals the weight in grams of 10,000 m. of yarn.

Reletter Paragraph (*f*) as (*h*) and delete the last sentence.

Reletter Paragraph (*g*) as (*i*) and change to read as follows:

(*i*) *Moisture Equilibrium for Testing.*—Yarn is considered to be in moisture equilibrium suitable for testing when after free exposure to standard laboratory air in motion for a period of 1 hr. the increase in weight of the sample does not exceed 0.1 per cent. For purpose of tests moisture equilibrium must be reached by absorption from a lower regain reached in an atmosphere having a relative humidity at least 15 per cent below that of the standard.

Section 14.—Change to read as follows:

14. The yarn shall be brought to a moisture equilibrium (Section 2(*i*)) prior to testing, and strength tests shall be made under the standard atmospheric conditions as prescribed in Section 2(*g*).

New Section.—Add the following as a new Section 17, renumbering the subsequent sections accordingly:

17. *Breaking Strength, Knot.*—(*a*) This method of test covers the determination of knot breaking strength of yarn. It is used as an approximate measure for "brittleness" of yarn. Elongation indicated in knot breaking tests is believed to be without significance. The results obtained by this method are the breaking loads required to rupture single strands having an overhand knot tied in the portion of the specimen length located between the clamps.

(*b*) *Test Conditions.*—The yarn shall be brought to a moisture equilibrium for testing (Section 2 (*i*)) prior to testing and knot strength tests shall be made under the standard atmospheric conditions as prescribed in Section 2(*g*).

(*c*) *Apparatus.*—The testing machine for determining knot breaking strength shall conform to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76). The pendulum type or the constant rate-of-load type may be used under the conditions specified in Section 15(*b*).

(*d*) *Test Specimens.*—The specimens of yarn for knot breaking strength shall be drawn from the side of the package instead of over the top, in order to avoid change in twist. A single overhand knot shall be tied at approximately the middle of each test length.

(*e*) *Procedure.*—An initial tension shall be applied to the specimen before gripping the specimen in the clamps. This tension shall be sufficient to remove any slack or loose portions of the knot, but not great enough to produce any appreciable elongation of the yarn.

Tests in which the specimens break at the edge of the jaws shall be rejected.

(*f*) *Report.*—The report shall include the following:

(*1*) Knot breaking strength expressed in grams, and

(*2*) Knot tenacity expressed in grams per denier or grams per grex.

Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 – 40 T):

As prepared by Subcommittee A-6, the following revisions are recommended by Committee D-13:

Section 3.—Change to read as follows:

3. Bleached wide cotton sheeting shall be classified in the following types on the basis of the number of warp yarns per inch plus the number of filling yarns per inch:

Type 200, combed percale.
Type 180, carded percale.
Type 140, heavy weight muslin.
Type 128, medium weight muslin.

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—PHYSICAL REQUIREMENTS OF BLEACHED WIDE COTTON SHEETING (FINISHED).

	Type 200	Type 180	Type 140	Type 128
Character of yarn...	combed	carded	carded	carded
Count, min., yarns per in.:				
Warp............	104	94	74	68
Filling............	98	84	66	60
Weight, oz. per sq. yd., min.......	3.6	3.6	4.6	4.2
Breaking Strength, min. lb. (Grab test):				
Warp............	65	60	70	55
Filling............	70	60	70	55
Sizing, max., per cent............	1.0	1.0	3.0	5.0

Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 – 40 T):

On the recommendation of Subcommittee B-1, Committee D-13 recommends an extensive addition to this method, comprising a new Procedure B by dispersions in cupriethylene diamine hydroxide, as appended hereto.[2]

Tentative Specifications for Mechanical Roll Felt (D 944 – 47 T):

The following revisions, prepared by Subcommittee A-10, are recommended by Committee D-13:

Section 3.—Change the heading to read "Types" by deleting the words "and Grades."

In Paragraph (*a*), delete the words "type 1 being the best" in the last line.

In the table in Paragraph (*b*), for classification 12R, change type "IX" to read "3X."

Section 4.—Change the first part of the sentence to read: "Roll felt is available in a standard range of thicknesses from $\frac{3}{64}$ to 1 in. in thickness . . ."

Table I.—Change the heading of the third column to read: "Wool Content, min., per cent," by deleting the word "fiber." Also change the felt number "17R1" to read "18R1."

In footnote *b* of this table, delete the word "fiber" from the first and last lines.

Tables II and IV.—Change the felt number "17R1" to read "18R1."

Table III.—Change the felt number "17R1" to read "18R1" and substitute the following values for nominal thickness in inches:

Nominal Thickness, in.	18R1	
	min.	max.
1/8	2.15	2.35
3/16	3.23	3.53
1/4	4.30	4.70
5/16	5.38	5.88
3/8	6.45	7.05
1/2	8.60	9.40

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 47 T):

The committee recommends that these definitions be revised by the deletion of certain terms regarding the flammability of fabrics, and the addition of new terms on combustible, noncombustible, inflammable and noninflammable fabrics. Revisions are also recommended in the definition of rayon, and rayon, modified, to be identical with these definitions as proposed above under revision of Methods D 258. Revisions are also recommended in the definitions of the terms plush, velvet, velveteen, and wool. New definitions are presented for monofilament, textile, standard yarn condition, sley, carded yarn, combed yarn, velour, and frieze. The revisions and additions to these definitions were prepared by Subcommittee B-2, and are appended hereto.[2]

ADOPTION OF TENATIVES AS STANDARD

The committee recommends that the tentative definitions for "azlon," "duck," and "fabric, woven narrow" be approved for reference to letter ballot of the Society for adoption as standard without revision and that they be incorporated in the Standard Definitions of Terms Relating to Textile Materials (D 123 – 47).

Tentative Methods of Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes (D 681 – 42 T):

The committee also recommends that these methods, with revisions prepared by Subcommittee A-5, as appended hereto,[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends that the definition of the term "glass" be deleted from the Standard Definitions of Terms Relating to Textile Materials (D 123 - 47) and that a note referring to the Standard Definition of the Term Glass (C 162 - 47) be added to the definition of "glass, textile." The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

---

[2] These revised methods and definitions were accepted by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Specifications for 0.007-in. Cotton Tape for Electrical Purposes (D 335 - 36):

Standard Method of Tests and Tolerances for Woolen Yarns (D 403 - 44):

Standard Method of Tests and Tolerances for Worsted Yarns (D 404 - 44):

Standard Specifications and Methods of Tests for Asbestos Yarns (D 299 - 42):

The committee recommends that the above standard methods and specifications be revised, as appended hereto,[2] and reverted to tentative. Subcommittee A-1 prepared the revisions of Standard Specifications D 335, Subcommittee A-3, those of Standard Methods D 403 and D 404, and Subcommittee A-4, those of Standard Specifications D 299.

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Felt (D 461 - 47):

The following proposed tentative revision of the new Section 2 is a modification of the tentative revision submitted in June, 1944. This revision was submitted by Subcommittee A-10.

New Section 2.—Delete the definitions for hardness and resilience.

Section 7 (*d*).—Change the first sentence to read as follows: "The presser foot shall be actuated by a freely-acting total deadweight load of 10 ± 0.5 oz. except that when by this method the felt has a density less than 0.08 oz. per cubic inch, the deadweight load shall be 2 ± 0.1 oz."

Standard Method of Test for Fiber Length of Wool (D 519 - 40):

On the recommendation of Subcommittee A-3, Section I on Wool, the following new Section 9 and Fig. 2 are recommended as a tentative revision of this standard:

> 9. *Cumulative Frequency Curve.*—It is recommended that a cumulative frequency curve be plotted from the data. As an illustration, Fig. 2 (the accompanying Fig. 1) has been constructed from the data given in Table I. For graphing the cumulative frequencies, the rectangular coordinate paper should have the vertical axis, the ordinate, divided into inch divisions, subdivided into ½-in. intervals over a distance of 7 in. sufficient to accommodate the longest fiber length. The horizontal axis, the abscissa should be divided into a total of 10 divisions each equivalent to 10 per cent and they in turn subdivided into 5 or 10 subdivisions. Any standard graph paper designated as "5 by 5" or "10 by 10" to the inch may be used. The points on the graph are plotted at the cell boundaries. For example, plotting the values found in Table I under the column headed "first cumulative less than," at 100 per cent the point is placed on the ordinate at 6.5 in., for 97.6 per cent at 6 in. and so on until 6.7 per cent at 0.5-in. interval is reached as shown in Fig. 2. For further information on plotting frequency curves, reference should be made to the A.S.T.M. Manual on Presentation of Data.

EDITORIAL CHANGES IN METHODS

The change in the definition for Rayon and the addition of the new definition for Estron will necessitate obvious editorial changes throughout the text and title of D 258 - 47 T, as well as in the following companion Standards:

Methods of Test for:

Rayon Staple (D 540 - 44), and Tolerances for Spun Rayon Yarns and Threads (D 507 - 44),

Strength of Rayon Woven Fabric When Wet (D 415 - 38),

Maximum Residual Shrinkage of Silk and Rayon Woven Fabrics (D 416 - 39),
Colorfastness of Dyed or Printed Wool, Silk, or Rayon Fabrics to Laundering or Domestic Washing (D 436 - 37),
Colorfastness of Dyed Cellulose Acetate Rayon to Atmospheric Fumes (D 682 - 47 T), and
Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 42).

Tentative Methods of Quantitative Analysis of Textiles (D 629 - 46 T):

Section 11 (*b*).—In the first and fourth sentences, delete "(100 times the weight of the specimen)." This is an erroneous statement since neither of the sulfuric acid solutions referred to has a specific gravity of 1.00.

Section 17 (*d*).—Delete the last sentence and substitute the following: "As the average diameters of vegetable fibers may vary over fairly wide limits, the average diameters of the fibers in the fabric being analyzed should be determined if accurate results are to be obtained. If maximum accuracy is not required, the composition may be calculated by using the data in Table II.

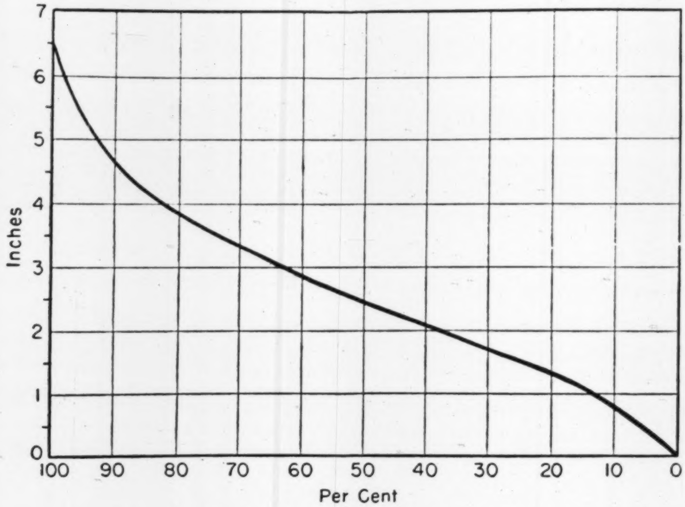


FIG. 1.—Cumulative Length Frequency Curve.

REPORT OF COMMITTEE D-14*

ON

ADHESIVES

Committee D-14 on Adhesives held two meetings during the past year. A two-day meeting was held in Cleveland, Ohio, on October 9 and 10, 1947. At this session a talk entitled "Some Factors Affecting Adhesive Bond Formation"[1] was given by H. P. Meissner of the Massachusetts Institute of Technology. A three-day meeting was held at Washington, D. C., on March 3 to 5, 1948 in conjunction with the spring Group Committee Meetings of the Society. At this meeting a talk entitled "Fundamental Properties of the Adhesion of High Polymers to Cellulose" was given by A. D. McLauren of the Polytechnic Institute of Brooklyn.

At present, Committee D-14 consists of 95 members of whom 30 are classified as consumers, 49 as producers, and 16 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Frank W. Reinhart.
Vice-Chairman, L. P. Hart, Jr.
Secretary, G. W. Koehn.
Assistant Secretary, J. H. Wills.

A Publicity Committee has been appointed by the chairman to encourage the publication of work carried on by various subcommittees and to see that the committee obtains a desirable amount of publicity.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Impact Strength of Adhesives (D 950 - 47 T). This recommendation was accepted by the Standards Committee on October 31, 1947, and the new tentative appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status. These methods were reviewed at the March, 1948, meeting of the committee, and in each case it was decided to continue these methods without revision so that further evaluation work may continue.

Tentative Methods of Test for:
Resistance of Adhesive Bonds to Chemical Reagents (D 896 - 46 T),
Tensile Properties of Adhesives (D 897 - 46 T),
Peel or Stripping Strength of Adhesives (D 903 - 46 T), and
Tentative Recommended Practice for:
Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (D 904 - 46 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman) continues to be very active. The Section on Tensile Strength has found that the present Tentative Method of Test for Tensile Properties of Adhesives (D 897 - 46 T) is a practical and usable one for other materials including metals. Present activities are devoted to developing a simplified method of tension testing. The Section on Shear Strength has concluded round-robin tests and submitted to letter ballot a proposed method for metal to metal test. The test method prepared by the Section on Impact Strength has been accepted by the full committee. A suggested method for fatigue testing is being prepared. Round-robin tests are being continued on flexural test specimens. Studies are being made on 90 deg. peel tests. A new method for cleavage testing of metal specimens will be submitted as tentative to the Administrative Committee on Standards in the near future.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] H. P. Meissner and E. W. Merrill, "Some Factors Affecting Adhesive Bond Formation," ASTM BULLETIN, No. 151, March, 1948, p. 80 (TP 88).

Subcommittee III on Permanence (R. F. Blomquist, chairman).—Mr. F. J. Wehmer has found it necessary to be relieved of his duties as chairman of this subcommittee and Mr. Blomquist took over as chairman after the October Meeting. Mr. Wehmer had been very active and his resignation was received with regret by the committee.

The Section on Effect of Moisture and Temperature has been working to establish satisfactory conditions for cyclic and continuous exposure test methods. The Section on the Effect of Chemical Factors is working on a second test method. The Section on the Effect of Light is making extensive round-robin tests. The Section on the Effect of Biological Factors has continued an extensive amount of testing and has accumulated considerable data as a basis for the preparation of a test method.

Subcommittee IV on Working Properties (C. B. Hemming, chairman).—The Section on Consistency has prepared a method of test for consistency for submission to the Administrative Committee on Standards in the near future. After preparation of adequate test methods, the Section on Coating and Spreading Rate has been relatively inactive. The Section on Penetration has been temporarily inactivated and its problems referred to the Subcommittee on Research. The Section on Tack and Blocking has been very active and a great deal of information has been accumulated for the development of a test method. The Section on Rate of Strength Development has explored the problem preliminary to the actual work of testing and preparation of test methods. A Section on Gap Filling Properties and one on Storage Working Life has been established.

Subcommittee V on Specifications (G. Reinsmith, chairman) has tentatively decided on specifications based on performance with the privilege of specifying the basic chemical type of material. On this basis specifications for adhesives suitable for adhering wood to wood are being prepared.

Subcommittee VI on Nomenclature (G. M. Kline, chairman) has prepared a list of terms and their definitions relating to adhesives which will be submitted to the Administrative Committee on Standards in the near future for publication as tentative.

Subcommittee VII on Research (A. G. H. Dietz, chairman) has been established during the past year to study fundamental adhesive problems. As this subcommittee has canvassed the main committee to obtain an expression on possible research projects a great many have been suggested but most replies favor work on pure strength properties. At the March meeting, a Section on Pure Shear Testing was established.

Subcommittee VIII on Electrical Properties (G. A. Olsen, chairman) was established at the March meeting to study the electrical properties of adhesives.

This report has been submitted to letter ballot of the committee, which consists of 95 members; 65 members returned ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. C. PLATOW,
Chairman.

L. P. HART, Jr.,
Secretary.

REPORT OF COMMITTEE D-15*
ON
ENGINE ANTIFREEZES

The current widespread interest in the development of more powerful and more efficient internal combustion engines has focused the attention of the engine designer as well as the ultimate consumer upon the need for maintaining the cooling system in peak operating condition. Recognition is now being given to the fact that the coolant must not only remain fluid under extremes of temperature but must also retard the corrosion of cooling system metals to keep the coolant passages clean and provide adequate dissipation of heat from the engine and radiator.

The Board of Directors of the Society, after studying the desirability of undertaking the preparation of test methods and standards for these important factors, authorized the organization of Committee D-15 on Engine Antifreezes. This committee held its organization meeting at Buffalo, N. Y., on May 26, 1947, at which time permanent officers were elected, a tentative scope of work was prepared, and specific projects were considered.

The scope, as adopted by Committee D-15 and approved by the Board, is as follows:

Scope.—The study of engine antifreezes, including terminology, identification and classification, methods of sampling and testing of engine antifreezes and cooling system corrosion inhibitors; interpretation and significance of tests; and the preparation of specifications.

The committee held its second meeting in Detroit, Mich., on January 13, 1948.

The Advisory Committee has held two meetings, one in Philadelphia, Pa., on July 8, 1947, and the other in Detroit, Mich., on January 13, 1948. At the first meeting, consideration was given to the formation of subcommittees to investigate the specific projects proposed by the main committee at its organization meeting. Of the seven subcommittees proposed, five have been organized and have held meetings. A summary of the work of the subcommittees is presented herewith.

At present there are 36 active members serving on the committee, of whom 17 are classified as producers, 9 as consumers, and 10 as general interest members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Freezing Point Determination (D. B. Brooks, chairman) has initiated a cooperative program of antifreeze sample testing to ascertain the accuracy and utility of the proposed methods for determining freezing point.

The National Bureau of Standards is verifying and redetermining data on solutions of methanol, ethanol, isopropanol, and ethylene glycol in connection with a revision of Letter Circular No. 28 on Antifreezes. This information will be available at the next meeting of the subcommittee. At that time, a program for such further work as is needed will be developed.

Subcommittee II on Antifreeze Field Testers (J. D. Klinger, chairman) has appointed a subgroup to develop performance specifications for hydrometers, outlining general requirements but not including details of manufacture. This group will make recommendations to the subcommittee at its next meeting.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

Subcommittee III on Physical Properties (H. S. Nutting, chairman) has circulated questionnaires on physical properties of antifreezes to members of the main committee. Based on the replies received, the subcommittee agreed to study the following factors or properties:

Appearance.—Color was not felt to be important unless federal regulations require a specific one. The presence or absence of sediment, it was agreed, was beyond the scope of an A.S.T.M. method or specification.

Boiling Point.—A procedure for the determination of boiling point is being prepared and will be submitted to the committee members for cooperative testing. A boiling point rather than a boiling range procedure was preferred.

Evaporation Rate.—This property was not believed to be of sufficient importance to develop a test method. Any procedure worked out, it was felt, would be very empirical.

Heat Transfer and Specific Heat.—These properties were considered to be of sufficient importance to warrant the development of test methods. A complete survey of existing test methods is being made. The results will be reported to the subcommittee at its next meeting.

Leakage and Creepage.—These properties were considered to be worthy of investigation. It was recommended, however, that the study be made by Subcommittee VI on Simulated and Actual Service Testing. Subcommittee VI has agreed to consider this matter at its next meeting.

Specific Gravity.—Work has been started on the preparation of an A.S.T.M. method for determining the specific gravity of packaged antifreeze. This subcommittee is cooperating with Subcommittee II on the determination of specific gravity of aqueous antifreezes.

Viscosity.—The recommendation was made that the Standard Method of Test for Kinematic Viscosity (D 445 - 46 T) be used as the test method and the results be reported in centistokes rather than centipoises to eliminate the need for determining density.

For the determination of viscosity at low temperatures (-40 F. or below) this subcommittee plans to await the recommendations of Subcommittee V of Committee D-2 on Petroleum Products and Lubricants which is currently investigating this phase of the problem.

Subcommittee IV on Chemical Properties (R. A. Willihnganz, chairman) has undertaken to establish generally accepted methods of test for determining the chemical properties of new and used antifreeze solutions. These properties include:

1. pH and reserve alkalinity,
2. Water content,
3. Ash and solids on evaporation, and
4. Detection, identification and composition.

Selected members of the group have prepared questionnaires on these subjects for submittal to the members of the main committee for comment.

This subcommittee feels that two problems with which it was charged, namely, storage stability, and cooling system gum and sludge formation, including anti-leak materials, are beyond its present scope and have asked the Advisory Committee to reconsider these assignments.

Subcommittee V on Effect of Antifreeze on Rubber Hose was authorized but not organized by the Advisory Committee at its first meeting. The possibility of forming a joint subcommittee with Committee D-11 on Rubber and Rubber-Like Materials is being explored.

Subcommittee VI on Simulated and Actual Service Testing (E. H. Keller, chairman) has initiated work on the two projects assigned to it by appointing subgroups to collect preliminary information and to report their findings to the subcommittee at its next meeting. These projects are:

1. The development of test methods for corrosion and foaming, and
2. The preparation of antifreeze and cooling system recommendations.

The subgroup on test methods has circulated a questionnaire to members of the main committee asking for recommended practices in testing antifreeze under laboratory and service conditions.

The Subgroup on Recommended Cooling Liquid Application has assembled available data on the subject and will hold a meeting to discuss the desirability of forming a joint study group with appropriate committees of the Society of Automotive Engineers, Coordinating Research Council, or the American Petroleum Institute for the release of the information collected.

Subcommittee VII on Specifications has been authorized but not organized by the Advisory Committee. This group will become active when the work of the other subcommittees has progressed further.

At the last meeting of the main committee, the present chairman, secretary and three of the four Advisory Committee members were reelected for the ensuing term of two years. By letter ballot, Mr. D. B. Brooks, a former Advisory Committee member, was elected Vice-Chairman, and Mr. J. C. Kratzer was elected an Advisory Committee member. Mr. Gus Kaufman was elected by voice vote at the meeting on June 25, 1948, to fill the vacancy left by Mr. Brooks on the Advisory Committee.

It was realized when Committee D-15 was formed that progress in attaining the desired objectives might be slow. In its first year of work, the committee has issued no tentative methods or specifications. However, all of the subcommittees are actively studying their assigned projects and much progress has been made.

This report has been submitted to letter ballot of the committee, which consists of 35 voting members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. R. WOLF,
Chairman.

E. H. KELLER,
Secretary.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES

Due to lack of progress on most phases of the committee's program of work, it was not possible to prepare a satisfactory report for last year. The committee has, however, remained active and held meetings at both the 1946 and 1947 Annual Meetings of the Society, both of which were well attended. The active membership has been slightly increased, giving the committee a voting membership of 41.

At the 1946 meeting, the committee, on the recommendation of the Advisory Subcommittee, placed its Subcommittee II on Crystallization of Rosin in a temporarily inactive status, it being reported by the chairman of that subcommittee that it had been impossible to devise any dependable test by which a tendency for any rosin to crystallize upon heating or after incorporation into any derived product, could be predetermined with any sufficient degree of accuracy and dependability.

S. R. Snider, of the Naval Stores Inspection Laboratory, Production and Marketing Administration, U. S. Department of Agriculture, was named as chairman of Subcommittee III on Volatile Oils in Rosin, replacing W. C. Smith, who found it impossible in his present position with the Procurement Division of the Treasury Department, to handle the collaborative work on the agenda of that subcommittee.

A comprehensive summary or tabulation of all known softening and melting point tests, the compilation of which had been started some years ago by C. E. Kinney, then chairman of Subcommittee I on Softening Point of Rosin, has been brought up to date by the present chairman of that subcommittee, J. M. Schantz. This tabulation is presented in an Appendix to this report.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, V. E. Grotlisch.
Vice-Chairman, J. E. Boyer.
Secretary, W. A. Kirklin.

TENTATIVES ADOPTED AS STANDARD

Tentative Definitions of Terms Relating to Naval Stores and Related Products (**D 804 - 46 T**).—These definitions have been widely circulated and given publicity in trade papers serving the naval stores industry and consumers of such products. Since no criticism or unfavorable comment has been received the committee recommends that these definitions be approved for reference to letter ballot of the Society for adoption as standard without revision

Tentative Methods of Sampling and Testing Dipentene (**D 801 - 46 T**).—On recommendation of Subcommittee VII, the committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

Section 9 (b).—Delete the last two sentences and substitute the following: "Observe and record the temperature when 5 ml. have been collected in the receiving cylinder, and thereafter when the level of the distillate reaches each

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

10-ml. division of the graduate, including a 95-ml. reading. Discontinue the distillation when the temperature reaches that specified for the minimum percentage requirement, or whenever the temperature rise stops and the thermometer reading starts to fall, and record the maximum temperature reached. Allow condenser to drain and record the percentage distilled."

Section 14.—Change to read as follows:

14. (*a*) *Distillation Method: Apparatus.*—The apparatus shall consist of the following:

(*1*) *Flask.*—A round bottom, shortneck, flask of 1000-ml. capacity.

(*2*) *Water Trap.*—A Dean and Stark moisture trap, 10-ml. capacity.

(*3*) *Condenser.*—A straight tube, water-cooled, glass reflux type, having a water jacket 400 mm. in length with an inner tube approximately 10 to 12 mm. in diameter.

(*4*) *Heat Source.*—A gas burner or an electric heater having suitable heat control.

(*5*) *Support for Flask.*—A piece of wire gauze resting on a ring support.

(*b*) *Procedure.*—Weigh 500 g. of dipentene into the flask. Connect the flask, trap and condenser with tight fitting, metal foil-covered corks. Insert a snug fitting burette cleaning brush into the top of the condenser, but capable of being lowered at will down to where the condensation of the distillate takes place. Apply heat and regulate it so that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per sec. Continue the distillation for 45 min. or longer until all moisture has been distilled from the flask. Avoid prolonged heating; 60 min. should be sufficient (Note). Bring down any droplets of water adhering to the walls of the condenser by adding a few drops of dipentene at the top of the condenser (enough to wet the brush) and then moving the brush up and down at the lower end of the condenser. Allow distillate to cool and disconnect flask and trap from condenser. Bring down any droplets of water that adhere to the walls of the trap with the aid of a glass stirring rod. Record the volume of water collected in the trap to the nearest 0.1 ml. Duplicate determinations should check within plus or minus 0.1 ml. of water collected, or 0.02 per cent.

(*c*) *Calculations.*—Calculate the moisture content as follows:

$$\text{Moisture, per cent} = \frac{\text{ml. of water}}{500} \times 100$$

NOTE.—The presence of oxidized constituents will cause high apparent moisture values due to their decomposition by heat. For old samples known to have been exposed to oxidation for a considerable length of time, as in stored partially filled containers, the alternate method referred to in Paragraph (*d*) should be used.

(*d*) *Alternate Method.*—If a more rapid determination is desired, or if the condition of the sample and the results obtained by the distillation method (Paragraph (*a*)) indicate oxidation, follow the procedure as described in the Tentative Method of Test for Water in Liquid Naval Stores (A.S.T.M. Designation: D 890).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction be continued in their present status pending further study by the various subcommittees.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—The chairman and several members have conferred with the chairman of Subcommittee B-5 of Committee D-4 on Road and Paving Materials, and with members of the Section on Softening Point of Committee E-1 on Methods of Testing, with respect to unification or combining the two present ring-and-ball softening point methods, Standard Methods D 36 – 26 for bituminous materials and Tentative Method E 28 – 42 T, for resins, asphalts and pitches. Progress is being made on this proposal. It is also hoped that a study of the various softening point methods outlined in the Appendix will be helpful in suggesting other approaches to the problem of determining softening point, the value or criterion for which depends entirely on the method used in making the test.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee III on Volatile Oils in Rosin (S. R. Snider, chairman).—Two samples of rosin, one of gum and one of wood rosin, prepared by compositing a large number of samples having abnormally high volatile oil content, and correspondingly low softening point, were prepared and sent to the members of this subcommittee for collaborative test of the Tentative Method of Test for Volatile Oil in Rosin (D 889 - 46 T). The work has not been completed and recommendations have not yet been made.

Subcommittee IV on Chemical Analysis of Rosin (W. D. Pohle, chairman) is continuing the study of the Tentative Method of Test for Acid Number of Dark Rosin (D 465 - 45 T) and of proposed methods for determining unsaponifiable matter, ash, and iron content of rosin. The chairman reports good agreement in some of these methods, and it is hoped to be able to recommend several of them for publication as tentatives after the Annual Meeting.

Subcommittee V on Tall Oil (A. Pollak, chairman) has continued to study the applicability of electrometric titration methods for determining the rosin acid number of crude tall oil. Several other features of the Tentative Methods of Testing Tall Oil (D 803 - 44 T) are also under study. It is hoped that sufficient progress can be reported to warrant revision of these methods and their adoption as standard by the next annual meeting of the Society.

Subcommittee VI on Pine Tar (J. P. Bain, chairman).—An A.S.T.M. standard thermometer[2] has been developed for use with the Stormer viscosimeter, used in determining viscosity of pine tar. This portion of the Tentative Methods of Test for Pine Tar (D 856 - 46 T) will now be resubmitted for collaborative test. Several variations of the method for determining acidity of pine tar have also been studied. The work has not yet progressed to permit a recommendation from the subcommittee.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman).—In addition to completing the study of the dipentene test methods, the subcommittee has been engaged in further study of the terpene alcohol determination in the Tentative Methods of Sampling and Testing Pine Oil (D 802 - 46 T). This was made desirable by the fact that an increase in the indicated content of terpene alcohol in pine oil was obtained when the distillation and catalytic dehydration were continued beyond the point where no further release of moisture from the distilling oil could be observed in the condenser. The causes for this increase in the quantity of recovered water, particularly whether it is derived from a delayed stage in the decomposition of the terpene alcohol, in the presence of the catalyst, are being sought. No report is as yet available. The subcommittee has also been working on standardization of two additional tests applicable to turpentine, namely, for acidity (resin acid content) and evaporation residue. These two test methods should be ready in the very near future, to permit their consideration by the main committee. When they are thus finally approved, they will in turn be submitted by Committee D-17 to Committee D-1 on Paint, Varnish, Lacquer, and Related Products for addition to the Tentative Methods of Sampling and Testing Turpentine (D 233 - 44 T). It was hoped to have these tests ready by the time Committee D-1 was required to take action on again adopting these methods as standard, but this was not possible. The acidity test is one of increasing interest, to producers, distributors, and to users of turpentine, as it has a bearing on the retention of condition, color and keeping quality of turpentine, particularly gum spirits of turpentine, after it has been packed in tin cans or shipped in galvanized steel drums.

---

[2] See A.S.T.M. Stormer Viscosity Thermometer 49C - 47T in Tentative Specifications for A.S.T.M. Thermometers (E 1 - 48 T), 1948 Supplement to Book of A.S.T.M. Standards, Part II.

Subcommittee IX on Definitions (J. L. Boyer, chairman) has completed the consideration of Tentative Definitions D 804, and has recommended the adoption as standard of the complete list of terms and definitions without further modification. No additional terms or proposed definitions are now under consideration.

This report has been submitted to letter ballot of the committee, which consists of 41 voting members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF SUBCOMMITTEE I ON SOFTENING POINT OF ROSIN

In 1943, Mr. C. E. Kinney, the chairman of Subcommittee I on Softening Point of Rosin, of Committee D-17 on Naval Stores was requested to collect and review the various methods in use for measuring the softening point or melting point of rosins and resins. While the "ball and ring" method (now A.S.T.M. Tentative Method E 28 – 42 T) had been standardized as a referee test, it was considered too time-consuming for routine testing and control use by a number of the users. A variety of different methods was and is being used by the many producers and consumers of rosins and resins, and it was thought that a study of all these methods would help in the standardization of a more rapid, better, and more generally acceptable method than the "ball and ring" test.

Various members of Committee D-17, as well as others interested in the subject, have submitted descriptions of their respective methods. In order to facilitate their comparison, these have been broken down uniformly into different sections such as bath system, essential apparatus, sample preparation, etc., and the features of each described separately.

Twenty-six different methods and modifications are described in the appended summary. Special consideration has been given to those methods which are related to buying and selling specifications. Undoubtedly, there are also other methods in use; the summary describes only those which have come to the attention of the subcommittee. With such a variety of methods in use, little imagination is required to visualize the confusion and duplicate testing being required by those consumers buying from different producers and by those producers selling to many different consumers, attempting to meet each of their several specifications.

The methods described in the summary are as follows:

I. A.S.T.M. Method of Test for Softening Point (*Ball and Shouldered Ring* Apparatus) (E 28 – 42 T)
II. Bakelite Modified A.S.T.M. *Ball and Ring Method* for Determining the Softening Point of Resins
III. Barber Asphalt Corporation Powder-Compression *Ball and Ring Method* for the Determination of the Softening Point of Gilsonite and Other High Melting Point Bitumens
IV. Rapid Method of Estimating the *Ring and Ball* Softening Point of Asphalts
V. Kuyer *Viscosity Method*
VI. Bureau of Chemistry and Soils (Smith) *Falling Ball Viscosity* Method for Determining the Viscosity of Rosin
VII. Kramer-Sarnow Softening Point Method as Applied to Bituminous Substances Fusing Above 176 F. (*Mercury Method*)
VIII. Kramer-Sarnow Softening Point Method for Bituminous Materials (*Mercury Method*)—As Standardized in Great Britain
IX. Kramer-Sarnow Softening Point Method for Bituminous Materials (*Mercury Method*)—As Modified by Nagel
X. Durran's *Mercury Method* for the Determination of the Melting Point of Resins
XI. Durran's *Mercury Method* for the Determination of the Melting Point of Resins—Modification of Wilber
XII. Durran's *Mercury Method* for the Determination of the Melting Point of Resins—Modification of Rangaswami (1929)
XIII. John D. Lewis (Hercules Powder Co.) *Mercury Method* for Determining the Softening Point of Resins

XIV. Strook and Wittenborg Corporation *Mercury Method* for Resin Melting Point Determinations

XV. Barber Asphalt Corporation *Mercury Surface Method* for Determining the Melting Point of Gilsonite and Similar Hydrocarbons

XVI. Newport Industries, Inc., *Capillary Tube Method* for Melting Point of Resins

XVII. Association Official Agricultural Chemists *Capillary Tube Method* for the Determination of the Softening and Melting Points of Resin

XVIII. Hercules *Capillary Tube Method* for the Determination of the Softening and Melting Points of Rosin

XIX. Hercules *Thermometer Drop Method* for Determining the Softening Point of Rosin

XX. General Electric *Thermometer Drop Method* for Determining the Melting Point of Bituminous Materials

XXI. *Drip Method* for the Determination of the Melting Point of Rosin

XXII. A.S.T.M. *Cube-In-Water Method* (D 61 – 38) for Pitches Having Softening Points Between 43 and 80 C.

XXIII. Johnson and Johnson *Cube-in-Air Method* for Rosin and Pitch

XXIV. *N.Y.T.L. Method* for the Determination of the Melting Point of Bituminous Substances

XXV. Dennis Melting Point Apparatus—*Electrically Heated Bar Method*

XXVI. Constant-Temperature Air Bath Modification of the A.S.T.M. *Ball and Ring Method* for Softening Point

No attempt has been made to summarize the good features of these methods or to criticize any bad features which they may have, it being considered better at this time to submit this tabulation to the committee as a whole for study and discussion. It is not believed that any one of the described methods would be acceptable for standardization by all the users for the variety of products which must be tested. The need for additional study combined with a considerable amount of cooperative work is obvious.

Respectfully submitted on behalf of the subcommittee,

J. M. SCHANTZ,
Chairman.

APPENDIX

SUMMARY OF SOFTENING POINT AND MELTING POINT METHODS USED FOR TESTING ROSINS AND RESINS

I. A.S.T.M. METHOD OF TEST FOR SOFTENING POINT (BALL AND SHOULDERED RING APPARATUS) (E 28 - 42 T)

Principle of Method:

Based on characteristic flow of sample within and from a ring, resulting from the action of a force equal to the combined weight of the sample and that of a superimposed steel ball under specified temperature conditions.

Bath System:

Consists of a liquid sample and heating bath using water as a heating medium for materials having a softening point below 80 C. and glycerine for those having a softening point above.

Apparatus:

Brass Shouldered Ring $\frac{23}{32}$ in. inside diameter above shoulder and $\frac{5}{8}$ in. below shoulder.

Steel Ball $\frac{3}{8}$ in. in diameter, weight 3.45 to 3.55 g.

Bath Container, a glass vessel not less than $3\frac{1}{2}$ in. in diameter and $4\frac{1}{8}$ in. in depth. (A 600-ml. low-form Griffin beaker meets requirements.)

Sample Preparation:

Sample, not less than 40 g., melted and portion poured into ring.

Cooled at room temperature for 20 min. if softening point is below 80 C. and 40 min. if above.

Test Procedure:

Sample and ball (not in position) immersed in test bath.

Bath cooled to not less than 45 C. and held at this temperature for 15 min.

With ball placed in position on sample in ring test, *bath temperature raised at uniform rate of 5 C. per min.*

Bath Agitation:

Heating bath agitated.

Softening Point (Meaning of Term):

Temperature of test bath at instant the softening sample, forced through ring by dropping ball, elongates sufficiently to touch bottom of beaker or plate 1 in. below ring.

Time Requirements:

Sample Preparation.—Melting, pouring, cooling sample to 45 C. and holding 15 min.—about 1 hr.

Test Procedure.—10 to 15 min.

Number of tests that can be made simultaneously with one heating bath—2.

Reproducibility:

Theoretical.—Duplicate determinations should not differ by more than 1 C. (1.8 F.).

Actual.—Duplicate determinations may differ by 2 C. (3.6 F.).

Reference:

A.S.T.M. Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 - 42 T), 1946 Book of A.S.T.M. Standards, Part II, p. 1679.

II. BAKELITE MODIFIED A.S.T.M. BALL AND RING METHOD FOR DETERMINING THE SOFTENING POINT OF RESINS

Principle of Method:

Same as for Method I.

Bath System:

Consists of an air sample bath within a liquid heating bath using glycerine as a heating medium.

Apparatus:

Steel Ring tapering from $\frac{11}{16}$ in. inside diameter at top to $\frac{3}{8}$ in. at bottom and $\frac{5}{32}$ in. in depth.

Steel Ball $\frac{5}{16}$ in. in diameter and weighing 1.97 to 2.07 g.

Heating Bath Container.—1500-ml. beaker.

Sample Bath Container.—Test tube $2\frac{1}{2}$ in. in diameter by 8 in. in length.

Sample Preparation:

Sample reduced to size of wheat grains or smaller and portion packed in ring and fused.

Test specimen cooled at room temperature.

Test Procedure:

Test tube for air bath immersed in heating bath.

Heating bath raised to temperature of 66 C. (150 F.) (simultaneous with sample preparation).

Ring with sample suspended in test tube.

Temperature of heating bath raised at a uniform rate of 4 to 5 F. (2 to 3 C.) per min.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of air sample bath at instant the softening sample, forced through the ring by falling ball, elongates sufficiently to touch plate $\frac{7}{8}$ in. below plate holding ring.

Time Requirements:

Sample Preparation.—Powdering sample, filling ring, and fusing sample as rapidly as possible—about 30 min.

Test Procedure.—Raising temperature of heating bath from 150 F. to the softening point at the rate of 4 to 5 F. per min.

Number of tests that can be made simultaneously with one heating bath—3.

Reproducibility:

Theoretical.—There should not be a variation greater than 0.83 C. (1.5 F.) in three tests run simultaneously.

Actual.—Unknown to author.

References:

Revised drawing of apparatus and description of method by the Bakelite Corp. (1931).

Navy Department Specifications 5205a for Ester Gum and 52R10b for Para-phenyl, phenolformaldehyde resin (1943).

III. BARBER ASPHALT CORPORATION POWDER-COMPRESSION BALL AND RING METHOD FOR THE DETERMINATION OF THE SOFTENING POINT OF GILSONITE AND OTHER HIGH MELTING POINT BITUMENS

Follow the A.S.T.M. Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 – 26)[1] or the A.S.T.M. Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 – 42 T),[2] with the following exceptions:

Sample Preparation:

Sample powdered and an excess transferred to the ring.

Excess pressed into ring by applying pressure in a suitable press or by hammering.

Reproducibility:

Theoretical.—Duplicate determinations should not differ by more than 5 F. (3 C.).

---

[1] 1946 Book of A.S.T.M. Standards, Part II, p. 605.

[2] *Ibid.*, p. 1679.

Actual.—Unknown to author.

Reference:

Description of method dated May 12, 1942, received from Barber Asphalt Co., April 21, 1944, courtesy of Mr. H. C. Howell, Chief Chemist.

IV. RAPID METHOD OF ESTIMATING THE RING AND BALL SOFTENING POINT OF ASPHALTS

Principle of Method:

Same as for Method I.

Bath System:

Consists of three constant temperature conditioning baths maintained at 37, 77, and 115 F. respectively, using water, and three constant temperature test baths maintained at 130, 180 and 230 F. respectively, to provide separate testing conditions for softening point ranges of 90 to 140 F., 135 to 185 F. and 180 to 250 F.

Apparatus:

Brass Ring $\frac{5}{8}$ in. in inside diameter and $\frac{1}{4}$ in. in depth.

Steel Ball $\frac{3}{8}$ in. in diameter, as specified in A.S.T.M. Softening Point Test for Bituminous Materials (D 36 – 26).[1]

Conditioning Bath Containers.—Glass or metal (seamless ointment boxes) substantially 2.17 in. (55 mm.) by 1.38 in. (35 mm.) as specified in the A.S.T.M. Standard Method of Test for Penetration of Bituminous Materials (D 5 – 25).[3]

Sample Test Bath Containers $6\frac{3}{4}$ in. in diameter by $5\frac{1}{2}$ in. in depth.

Sample Preparation:

Samples melted and portion poured into two rings (placed in Tobin bronze block) at a specified temperature for each range.

Samples in block cooled in air at room temperature for 1 min.

If in range 1 or 2 samples further cooled in 77 F. bath, and if in range 3 cooled in 115 F. bath, for 4 min.

Samples removed from block and if in range 1 or 2 cooled in 37 F. bath, and if in range 3 cooled in 115 F. bath, for 3 min.

Test Procedure:

Steel balls placed in position and sample, if in range 1, immersed in *constant temperature bath* at 130 F.; if in range 2, at 180 F.; and if in range 3, at 230 F.

Bath Agitation:

Each sample conditioning bath and test bath agitated.

Softening Point (Meaning of Term):

The *estimated temperature of the softening sample* at the instant that, forced through and from the ring by the falling ball, it elongates sufficiently to touch the bottom of the bath container, a distance of 1 in. below the ring. The elapsed time is taken from the moment the sample is immersed in the constant temperature bath, and the time-softening point relationship is obtained from the prepared conversion tables.

Time Requirements:

Sample Preparation.—Melting large sample, pouring into two rings, and cooling—about 30 min.

Test Procedure.—Raising temperature of sample to softening point in proper test bath—asserted to be about 10 min.

Reproducibility:

Theoretical.—Time in seconds should not differ by more than 7 per cent, based on average value.

Actual.—Unknown to author.

Reference:

A. B. Hersberger and C. Overbeck, "A Rapid Method of Estimating the A.S.T.M. Ring-and-Ball Softening Point of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 889 (1942).

V. KUYER VISCOSITY METHOD

Principle of Method:

Based on the characteristic flow of melted sample down the perpendicular side of a tube under specified constant temperature conditions.

---

[3] 1946 Book of A.S.T.M. Standards, Part II, p. 600.

Bath System:

Consists of an air sample bath or baths in a liquid heating bath using water as a heating medium.

Apparatus:

Sample Bath Containers.—Homeopathic vials 13.5 mm. in diameter and 75 mm. in length.

Circular Vial Holder consisting of two plates about 50 mm. apart with 12 matched peripheral recesses provided with spring clips for suspending vials in vertical position.

Heating Bath Container.—Metal can or any suitable container about 150 mm. in diameter and 250 mm. in depth with cover slotted to its center.

Sample Preparation:

Sample powdered and 4 g. weighed into each vial.

Stoppered vial inverted, that is with corks down, and immersed in heating bath of boiling water for 15 min.

Sample vials removed from heating bath and quickly reversed in holder.

Test Procedure:

After reversing in heating bath with water at boiling temperature, sample vials re-immersed for exactly 2 min.

Sample vials removed from water and distance of flow measured in $\frac{1}{30}$-in. units.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Degree of softening indicated by the *distance sample flows* down the side of vial in vertical position during a period of 2 min. at the temperature of boiling water.

Time Requirements:

Sample Preparation.—Powdering, weighing, and introducing into vial—about 10 min.

Heating at temperature of boiling water for 15 min.

Test Procedure.—2 min.

Number of tests that can be made simultaneously—12.

Reproducibility:

Theoretical.—Variation in tests within $\frac{5}{30}$ in.

Actual.—Unknown to author.

Reference:

"Determination of the Viscosity of Rosin" using the method devised by B. E. Kuyers, The Tanglefoot Co., Grand Rapids, Mich., dated January 20, 1928.

VI. BUREAU OF CHEMISTRY AND SOILS (SMITH) FALLING BALL VISCOSITY METHOD FOR DETERMINING THE VISCOSITY OF ROSIN

Principle of Method:

Based on the fall of a steel ball through the melted sample held at a specified constant temperature.

Bath System:

Consists of a cluster of sample containers within a liquid heating bath using boiling water as a heating medium.

Apparatus:

Sample Containers—Viscosity Tubes.—Test tubes 15 mm. ($\frac{5}{8}$ in.) and 125 mm. (5 in.) in length marked with line 75 mm. (3 in.) from bottom.

Heating Bath Container.—1500-ml. low-form beaker.

Circular Tube Holder consisting of two parallel plates 100 mm. apart provided with matched openings in periphery for receiving tubes and opening in center for receiving glass receptacle for viscosity balls, the plates being of suitable dimensions to permit suspending of assembly in the heating bath with the upper plate acting as cover.

Steel Balls 0.25 in. in diameter.

Sample Preparation:

Sample powdered and lightly packed in viscosity tubes within $\frac{1}{2}$ in. of top.

Sample melted by immersing tubes, stoppered with solid cork, in the boiling water heating bath and brought up to mark by adding more sample. Heating continued for 30 min. to bring sample, holder, and balls to the temperature of the heating bath, that is, to the boiling point of water.

Test Procedure:

Without removing viscosity tube assembly from the heating bath, replace solid cork with cork fitted with glass guide tube adjusted to 5 mm. above surface of the sample.

Remove one of the viscosity balls from the glass ball receptacle which has been kept immersed in the heating bath and immediately drop through the guide tube.

Bath Agitation:

No bath agitation.

Softening Point (Meaning of Term):

Time in seconds *for ball*, after release, *to fall through sample* at temperature of boiling water in viscosity tube, a distance of 75 mm. (3 in.).

Time Requirements:

Preparation of Sample.—Powdering and packing sample in viscosity tubes, melting, bringing up to mark, and heating for 30 min.—about 60 min.

Test Procedure.—Adjusting glass slide and dropping viscosity ball—6 to 50 sec.

Number of tests that can be made in rapid succession with one heating bath—8.

Reproducibility:

Theoretical.—No statement made.

Actual.—Wide variance in 1928 cooperative tests.

Reference:

Description and instructions furnished by the Bureau of Chemistry and Soils for cooperative laboratory tests conducted by the A.S.T.M. Committee D-17 on Naval Stores in 1928.

VII. KRAMER-SARNOW SOFTENING POINT METHOD AS APPLIED TO BITUMINOUS SUBSTANCES FUSING ABOVE 176 F. (MERCURY METHOD)

Principle of Method:

Based on the characteristic flow of a sample in and from a vertical open tube into a heating bath liquid, resulting from the action of a force equal to the combined weight of the sample and a superimposed layer of mercury under specified temperature conditions.

Bath System:

Consists of a liquid heating bath using water as a heating medium for samples melting below 176 F. and castor oil for those above.

Apparatus:

Sample Holders.—Glass tubes with open ends, 6 to 7 mm. ($\frac{1}{4}$ in.) in inside diameter and about 80 mm. ($3\frac{1}{8}$ in.) in length, and one glass tube of same dimensions with one closed end containing thermometer and sufficient mercury to cover bulb.

Heating Bath Container.—Beaker of suitable dimensions.

Sample Preparations:

Sample powdered, portion pressed into lower end of tube, softened with gentle heat, and packed with snug-fitted glass rod to form a plug 7 to 9 mm. in length.

Cooled and end of plug scraped out until exactly 5 mm. remains, leaving a 2 to 4-mm. air space between the plug and lower end of tube.

A 5-g. portion of mercury, corresponding to 10 to 11 mm. of mercury, introduced on top of plug.

Test Procedure:

Sample holders containing samples loaded with mercury and thermometer well, passing through and suspended from beaker cover, are immersed in heating bath that has been adjusted to a temperature at least 25 C. below the estimated softening point of the sample.

Temperature of the heating bath raised at the uniform rate of 4 C. per min.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of the heating bath as registered by thermometer in mercury-filled tube immersed in heating bath.

Time Requirements:

Preparation of Sample.—Powdering, pressing into melting point tube, cooling, scraping out portion of sample at end of tube, and weighing and introducing mercury—estimated about 20 min.

Test Procedure.—Raising temperature of mercury layer to softening point at rate of 4 C. per min.—about 10 min.

Number of tests that can be made simultaneously with one bath—4.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

References:

Originally proposed by G. Kramer and C. Sarnow in German periodical of 1890. Method as given is taken from "Improved Instruments for the Physical Testing of Bituminous Materials" by Herbert Abraham, *Proceedings*, Am. Soc. Testing Mats., Vol. 9, p. 575 (1909), and Vol. 11, p. 673 (1911); also H. Abraham, "Asphalt and Allied Substances," Methods of Testing, Fifth Edition, Vol. 2 (1905).

VIII. KRAMER-SARNOW SOFTENING POINT METHOD FOR BITUMINOUS MATERIALS (MERCURY METHOD)—AS STANDARDIZED IN GREAT BRITAIN

Principle of Method:

Based on the characteristic flow of a sample in and from a vertical open tube into air, resulting from the action of a force equal to the combined weight of the sample and a superimposed layer of mercury under specified temperature conditions.

Bath System:

Consists of an air sample bath container suspended within a heating bath container so that the tops of the two containers are in the same plane and the circular open space between them sealed to form a jacket, castor oil being used as a heating medium.

Apparatus:

Sample Holders.—Brass or stainless steel tubes 6.5 mm. ($\frac{1}{4}$ in.) in inside diameter and 5 mm. ($\frac{3}{16}$ in.) in length.

Mercury Holders.—Glass tubes with open ends 6.5 mm. ($\frac{1}{4}$ in.) in inside diameter and 95 mm. ($3\frac{3}{4}$ in.) in length.

Thermometer Container or Well.—Same as glass mercury tubes except closed at one end and containing sufficient mercury to surround the bulb.

Sample Preparation:

Sample heat-softened and portion molded into sample holder (actual method not disclosed).

Cooled and holder connected, as an extension to the mercury tube by a short rubber tube.

Sample loaded with 5 g. of mercury from pipet, equivalent to a mercury height of 10 to 11 mm.

Test Procedure:

Sample holders with mercury tubes attached and thermometer wells with thermometer, passing through and supported by metal cover resting on the top of both beakers, are suspended in the air bath.

Temperature of air bath is registered by thermometer in mercury-filled thermometer well, *raised at uniform rate of 4 C. per min.*

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of the sample air bath at the instant that the softening sample flows from the holder sufficiently to permit the mercury to break through.

Time Requirements:

Sample Preparation.—Melting sample, introducing into holder, cooling, attaching holder to mercury tube, weighing mercury into tube—estimated 20 to 30 min.

Test Procedure.—Sample holder assembly suspended in heating bath and temperature raised to softening point at rate of 4 C. per min.—estimated 10 to 15 min.

Number of tests that can be made simultaneously with one bath—4.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

Reference:

H. Abraham, "*Asphalts and Allied Substances*," Fifth Edition, Vol. 2, pp. 1069–1070 (1945).

IX. KRAMER-SARNOW SOFTENING POINT METHOD FOR BITUMINOUS MATERIALS (MERCURY METHOD)—AS MODIFIED BY NAGEL

Principle of Method:

Same as for Method VIII.

Bath System:

Consists of an air sample bath within a liquid heating bath using oil or glycerine as a heating medium.

Apparatus:

Sample Holder.—Glass tube open at both ends, 5-mm. ($\frac{3}{16}$-in.) inside diameter reduced to 3 mm. ($\frac{1}{8}$ in.) beginning at a distance of 10 mm. ($\frac{3}{8}$ in.) from one end (total length not given).

Air Sample Bath Container.—Test tube of suitable dimensions to receive sample holder and thermometer.

Heating Bath Container.—Beaker of suitable dimensions to receive sample bath assembly.

Sample Preparation:

Sample finely powdered, portion introduced into large diameter end and packed into small diameter end until entire length of construction is filled to form a plug 10 mm. in height.

Covered with 5 g. of mercury, equivalent to a height of 19 mm. ($\frac{3}{4}$ in.).

Sample holder containing sample loaded with mercury, and thermometer, passing through and supported by stopper, are suspended in test tube.

Test Procedure:

Test tube assembly immersed in heating bath.

Temperature of air bath raised rapidly to within 25 C. of softening point.

Temperature held stationary for 15 min.

Temperature of air bath raised at uniform rate of 1 C. per min.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of air bath at the instant that the softening samples flow from the holder sufficiently to permit the mercury to break through.

Time Requirements:

Sample Preparation.—Sample powdered and packed in holding tube, and mercury weighed and introduced into tube—estimated about 10 min.

Test Procedure.—Temperature air bath raised to within 25 C. of softening point, held stationary for 15 min., and raised at rate of 1 C. per min.—estimated about 45 to 50 min.

Number of tests that can be made simultaneously with one heating bath—1 or more.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

References:

Published in German periodical in 1925 and 1926. In "Physical and Chemical Examination of Paints, Varnishes, Lacquers, Colors," by Gardner, Sixth Edition, p. 719 (1933). Also Technical Paper No. 4, "Fundamental Physical Properties of Lac," Part II, Thermal Properties.

X. DURRAN'S MERCURY METHOD FOR THE DETERMINATION OF THE MELTING POINT OF RESINS

Principle of Method:

Based on the characteristic upward flow of sample through a superimposed layer of mercury of definite weight, under specified temperature conditions.

Bath System:

Consists of a mercury sample bath within a liquid heating bath, using sulfuric acid as a heating medium.

Apparatus:

Sample Bath Container.—Test tube 17 mm. ($\frac{5}{8}$ in.) in diameter and 150 mm. (6 in.) in length.

Heating Bath Container.—Beaker of suitable dimensions.

Sample Preparation:

Two grams of sample weighed into test tube and fused by immersing tube in heating bath and raising temperature of bath until sample is melted.

Test tube, with thermometer suspended in melted sample, removed and sample allowed to cool.

Solid sample covered with 50 g. of mercury, equivalent to a layer 16.2 mm. ($\frac{5}{8}$ in.) in height.

Test Procedure:

Test tube with sample and mercury reimmersed in heating bath after adjusting temperature to 30 C. below softening (melting) point.

Temperature of sample and mercury raised at uniform rate of 12 C. per min.

Bath Agitation:

Heating bath agitated.

Melting Point (Meaning of Term):

Temperature of sample at instant that the softening sample forced upward by the sagging mercury, breaks through the surface of the mercury.

Time Requirements:

Sample Preparation.—Weighing, fusing, and cooling sample and covering sample with mercury—estimated about 20 min.

Test Procedure.—Reimmersing in heating bath, adjusting bath to 30 C. below melting point, and raising temperature at rate of 2 C. per min.—estimated about 20 to 30 min.

Number of tests that can be made simultaneously with one heating bath—1 or more.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

Reference:

T. H. Durrans, "A New Method for the Determination of the Melting Point of Resins," *Journal*, Oil and Colour Chemists Assn., Vol. 12, pp. 173–175 (1929). (First reported in 1928.)

XI. DURRAN'S MERCURY METHOD FOR THE DETERMINATION OF THE MELTING POINT OF RESINS—MODIFICATION OF WILBER

Principle of Method:

Same as for Method X.

Bath System:

Consists of a mercury sample bath heated by means of a sand bath.

Apparatus:

Sample Bath Container.—Porcelain crucible, No. 1 low-form, 46 mm. ($1\frac{13}{16}$ in.) in diameter at top and 29 mm. ($1\frac{1}{8}$ in.) in height, with a capacity of 30 ml.

Sand Bath.—Cup of Cleveland flash tester, 67 mm. ($2\frac{11}{16}$ in.) in diameter and 31 mm. ($1\frac{1}{4}$ in.) in height.

Sample Preparation:

Sample ground to about the fineness of granulated sugar, and 2.0 g. weighed into crucible and melted with the least possible heat, making a calculated resin layer 4.75 mm. in height.

Cooled to room temperature and covered with exactly 292 g. of mercury, making a calculated layer of mercury 22.5 mm. in height.

Thermometer suspended so that bulb is completely immersed in the mercury.

Test Procedure:

At the end of 10 min., sample and mercury are heated so that temperature of mercury rises at the rate of 3 C. per min.

When sample melts, *temperature of mercury is raised at the rate of 2 C. per min.*

Bath Agitation:

No agitation.

Melting Point (Meaning of Term):

Temperature of the mercury at the instant the softening sample, forced upward by the mercury, breaks through the surface of the mercury.

Time Requirements:

Sample Preparation.—Weighing, fusing, cooling of sample, and covering with mercury—estimated about 30 min.

Test Procedure.—Raising temperature at rate of 2 C. per min. from room temperature—20 to 45 min.

Number of tests that can be made simultaneously—1.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

Reference:

Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Eighth Edition, p. 326 (1939).

XII. DURRAN'S MERCURY METHOD FOR THE DETERMINATION OF THE MELTING POINT OF RESINS—MODIFICATION OF RANGASWAMI[4] (1929)

Principle of Method:

Same as for Method X.

Bath System:

Consists of a mercury sample bath heated directly on a wire gauze by a micro burner.

Apparatus:

Sample Bath Container.—Porcelain crucible, 40 mm. ($1\frac{9}{16}$ in.) in diameter and 25 mm. (1.0 in.) in height with a capacity of 17 ml.

Sample Preparation:

A 9.29 g. portion of sample weighed into crucible and gently heated until melted making a calculated layer 0.8 mm. in height.

Cooled at room temperature and covered with 25 g. of mercury making a calculated layer 5.9 mm. in height.

Thermometer suspended so that bulb is immersed in mercury.

Actual Test:

Temperature of the mercury (and sample), protected from drafts, *raised at the uniform rate of 2 C. per min.*

Bath Agitation:

No agitation.

Melting Point (Meaning of Term):

Temperature of mercury at the instant that the softening sample forced upward by the mercury breaks through the surface of the mercury.

Time Requirements:

Sample Preparation.—Weighing, fusing, and cooling sample and covering with mercury—estimated about 15 min.

Test Procedure.—Heating at rate of 2 C. per min. from room temperature—30 to 60 min.

Number of tests that can be made simultaneously—1.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

Reference:

M. Rangaswami, "A Note on the Determination of the Melting Point of Resins," *Journal*, Oil and Colour Chemists Assn., Vol. 13, p. 287 (1930).

---

[4] M. Rangaswami, Biochemical Laboratory, Indian Sae Research Institute.

XIII. JOHN D. LEWIS (HERCULES POWDER CO.) MERCURY METHOD FOR DETERMINING THE SOFTENING POINT OF RESINS

Principle of Method:

Based on the characteristic upward flow of sample through a superimposed layer of mercury of definite volume under specified temperature conditions.

Bath System:

Consists of a mercury sample bath within a liquid heating bath using glycerine or mineral oil as a heating medium.

Apparatus:

Sample Bath Containers.—Melting point tubes 0.31 mm. ($\frac{5}{16}$ in.) in inside diameter and 3 mm. in length, with mark etched around tube 1.0 in. above point where rounded bottom resumes tangential with side wall.

Heating Bath Container.—600-ml. beaker.

Sample Preparation:

Sample, 0.2 g. preferably in one piece, weighed into melting point tube and fused.

Cooled at room temperature and covered with layer of mercury up to mark.

Test Procedure:

Heating bath cooled to at least 30 C. below expected softening point.

Melting point tube containing sample immersed in heating bath and *temperature raised at uniform rate of 4 C. per min.*

Bath Agitation:

Heating bath agitated.

Softening Point (Meaning of Term):

Temperature of heating bath at the instant the softening sample, forced upward by the sagging mercury, reaches the mark.

Time Requirements:

Sample Preparation.—Weighing and fusing sample and filling tube up to mark—about 10 min.

Test Procedure.—Cooling heating bath to at least 30 C. below softening point of sample and raising temperature at rate of 4 C. per min. to softening point—10 to 20 min.

Number of tests that can be made simultaneously with one heating bath—1 or more.

Reproducibility:

Theoretical.—No statement made.

Actual.—About 1 C.

Reference:

"Softening Point—Mercury Method," from "Manual of Analytical Methods—Revision of 1942," Hercules Powder Co.

XIV. STROOK AND WITTENBORG CORPORATION MERCURY METHOD FOR RESIN MELTING POINT DETERMINATIONS

Principle of Method:

Same as for Method X.

Bath System:

Consists of a series of mercury sample baths within a liquid heating bath using, preferably, Russian white oil as a heating medium.

Apparatus:

Sample Bath Containers.—Six test tubes $\frac{5}{8}$ in. (16 mm.) in diameter and 6 in. (152 mm.) in length.

Heating Bath Container.—Glass jar 6 in. (152 mm.) in diameter and $9\frac{1}{4}$ in. (235 mm.) in length with segment cut out longitudinally to make an opening $2\frac{3}{8}$ in. (68 mm.) in width and $8\frac{3}{4}$ in. (222 mm.) in length. Assembled in horizontal position. Insulated.

Sample Preparation:

Sample powdered and 3 g. fused in test tube.

Cooled at room temperature and covered with layer of 50 g. of mercury, equivalent to a layer of 18 mm. ($\frac{23}{32}$ in.) in depth. Thermometer passing through and suspended by cork, inserted in test tube with bulb in mercury layer.

Test Procedure:

Heating bath temperature raised to

within 20 to 30 C. of the melting (softening) point of that sample having the lowest melting point.

Tube or tubes containing sample and mercury immersed in *heating bath* and bath *raised at uniform rate of 3 C. per min.*

Bath Agitation:

Heating bath agitated.

Melting Point (Meaning of Term):

Temperature of the mercury at the instant that the softening sample forced upward by the sagging mercury breaks through the mercury.

Time Requirements:

Sample Preparation.—Powdering, weighing, fusing in test tube, cooling, and covering with layer of mercury—estimated 15 to 20 min.

Test Procedure.—Starting 30 C. below expected melting point at rise of 3 C. per min.—10 to 20 min.

Number of tests that can be made simultaneously with one bath—1 to 6 or 8.

Reproducibility:

Theoretical.—No statement made.

Actual.—Findings by Strook and Wittenborg Corp. are that individual determinations in same bath check to within 1 C. and between baths to within 2 C. for melting point ranges up to 148 C.

Reference:

"Improved Control in Method for Resin Melting Point Determinations," October, 1940, issue of "Resinews" published by Strook and Wittenborg Corp., 60 E. 42nd St., New York, N. Y.

XV. BARBER ASPHALT CORPORATION MERCURY SURFACE METHOD FOR DETERMINING THE MELTING POINT OF GILSONITE AND SIMILAR HYDROCARBONS

Principle of Method:

Based on the flow of the rough surfaces of sample particles under specified temperature conditions, producing a change in the appearance of the sample.

Bath System:

No bath system. Mercury in a suitable container used as a sample heating medium and a means of floating the powdered sample.

Apparatus:

Mercury Container.—A 3-oz. ointment box, approximately 2.0 in. (50 mm.) in diameter and 2½ in. (63 mm.) in depth. A Gill style ointment box (tin) is suitable. A 2½-in. watch glass with $\frac{5}{16}$-in. (8-mm.) hole drilled in center.

Magnifying Glass of 4.0 to 6.5 diameters.

Sample Preparation:

Sample ground and sieved to size, passing No. 20 sieve but held on No. 30.

Five to fifteen particles placed on cover glass and floated at room temperature on surface of mercury having a depth of 3 cm. ($1\frac{3}{16}$ in.).

Container covered with watch glass, convex side up, and thermometer suspended through glass and immersed in mercury.

Test Procedure:

Mercury heated from room temperature until a temperature of 200 to 212 F. is reached and then *at a uniform rate of 10 to 15 F. per min.*

Particles observed through magnifying glass.

Bath Agitation:

Not applicable.

Melting Point (Meaning of Term):

Temperature of mercury and sample at instant all sharp edges and other lines and marks which defined the original shape or structure of the particles round out and disappear when viewed under a magnification of approximately 6 diameters.

Time Requirements:

Sample Preparation.—Grinding, screening, placing particles on cover glass, and floating cover glass on mercury—estimated about 5 min.

Test Procedure.—Raising temperature

from room temperature to 200 to 220 F. (or adjusted temperature) at the rate of 30 F. per min. and then to the melting point at the rate of 10 to 15 F. per min.—estimated about 10 min.

Number of tests that can be made simultaneously without duplicate equipment—1.

Reproducibility of Results:

Theoretical.—Not stated.

Actual.—Unknown to author.

References:

Description by Barber Asphalt Co., Mauner, N. J., November 28, 1930. Also, in Technical Paper No. 4, "Fundamental Physical Properties of Lac," Part II, "Thermal Properties," London Shellac Research Bureau.

XVI. NEWPORT INDUSTRIES, INC., CAPILLARY TUBE METHOD FOR MELTING POINT OF RESINS

Principle of Method:

Based on the characteristic change in appearance of a powdered or crystalline sample as it progresses from the solid to liquid state under specified temperature conditions.

Bath System:

Consists of glass tube sample containers immersed in liquid heating bath using glycerine to same level in both beakers.

Apparatus:

Sample Containers.—Capillary tubes, glass, $\frac{1}{8}$-in. (1.5-mm.) bore, overall length $2\frac{11}{16}$ in. (68 mm.), with tip drawn out and sealed at end $\frac{7}{8}$ in. (22 mm.):

Heating Bath Container.—Inside beaker of 150-ml. capacity and outside beaker of 600-ml. capacity, with perforated ring resting on rim or large beaker supporting the small beaker.

Thermometer of suitable range, carefully standardized, graduated to 0.2 C. or 0.5 F.

Sample Preparation:

Sample pulverized to fine uniform powder and packed into capillary tubes to a depth of 1 in. (25 mm.) by tapping.

Tubes containing samples attached to thermometer with rubber bands so that closed end of tube is level with bottom of thermometer bulb.

Test Procedure:

Thermometer with attached sample tubes suspended in small inner beaker with tubes submerged to within $\frac{1}{4}$ in. (6 mm.) of their tops.

Heating bath temperature raised 1 C. per min.

Bath Agitation:

None specified.

Melting Point (Meaning of Term):

The *temperature at which* the powdered *sample begins to fuse* together and stick to the side of the glass.

Time Requirements:

Sample Preparation.—Powdering and packing samples into melting point tubes and attaching tubes to thermometer—estimated about 10 min.

Test Procedure.—Immersing thermometer and tubes in heating bath, and raising temperature 1 C. per min.—30 to 60 min.

Reproducibility:

Theoretical.—No statement made.

Actual.—Unknown to author.

Reference:

Newport Industries, Inc., publication.

XVII. ASSOCIATION OFFICIAL AGRICULTURAL CHEMISTS CAPILLARY TUBE METHOD FOR THE DETERMINATION OF THE SOFTENING AND MELTING POINTS OF RESIN

Principle of Method:

Same as for Method XVI.

Bath System:

Consists of an air sample bath within

a liquid heating bath using glycerine as a heating medium.

Apparatus:

Sample Containers.—Four melting point tubes, $\frac{1}{16}$ in. (15 mm.) in inside diameter and 2 in. (50 mm.) in length, closed at one end.

Thermometer.—Used as a holder for melting point tubes.

Air Sample Bath Container.—Test tube $\frac{3}{4}$ in. (20 mm.) in diameter and 6 in. (150 mm.) in length.

Heating Bath Container.—300-ml. Erlenmeyer flask with neck 1 in. (25 mm.) in diameter.

Sample Preparation:

Sample of 1 to 2 g. powdered and a portion lightly packed in four melting point tubes to a depth of about 10 mm.

Two tubes attached to thermometer opposite bulb.

Thermometer with attached tubes suspended by cork in air bath test tube.

Test Procedure:

Air bath container (test tube) with melting point tube assembly immersed in heating bath which has been brought to a temperature of 45 C.

Temperature of air bath above 50 C. *raised at uniform rate of 1 C. per min.*

Bath Agitation:

No agitation.

Softening Point and Melting Point (Meaning of Terms):

Softening Point.—The *temperature* at which the powdered rosin in a melting point tube begins to *darken and coalesce.*

Melting Point.—The *temperature* at which the powdered rosin in a melting point tube loses its powdered or crystalline appearance and becomes wholly *transparent.*

Time Requirements:

Sample Preparation.—Powdering sample, filling four melting point tubes, and attaching tubes to thermometer—about 10 min.

Test Procedure.—Raising sample bath to softening point—10 to 20 min.

Number of tests that can be made simultaneously with one heating bath—2.

Reproducibility:

Theoretical.—Four tests must not vary more than 1 C.

Actual.—Unknown to author.

References:

Instructions for the determination of the softening and melting points of resin—A.O.A.C. Referee (1924).

Instructions for the determination of the softening and melting points of rosin—Cooperative laboratory tests conducted by A.S.T.M. Committee D-17 (1928).

XVIII. HERCULES CAPILLARY TUBE METHOD FOR THE DETERMINATION OF THE SOFTENING AND MELTING POINTS OF ROSIN

Principle of Method:

Same as for Method XVI.

Bath System:

Consists of glass tube sample containers immersed in a liquid heating bath using water or a suitable oil as a heating medium.

Apparatus:

Sample Containers.—Melting point tubes, approximately $\frac{1}{8}$ in. (1.5 mm.) in inside diameter and 4 in. (100 mm.) in length, closed at one end.

Heating Bath Container.—Any suitable container.

Thermometer.—Used as holder for melting point tubes.

Sample Preparation:

Sample powdered and portion lightly packed into at least two melting point tubes to a depth of about $\frac{3}{8}$ in. (10 mm.).

Tubes, containing samples, attached to the thermometer.

Test Procedure:

Melting point tube assembly suspended

so that tubes and thermometer bulb are immersed in the heating bath.

Heating bath temperature raised rapidly to within 15 C. of softening point of samples, that is, to about 45 C. for ordinary rosins.

Temperature raised at the uniform rate of 1 C. per min.

Bath Agitation:

No agitation.

Softening and Melting Points (Meaning of Terms):

Softening Point.—The *temperature of the heating bath* at the instant that the sample *darkens and coalesces.*

Melting Point.—The *temperature of the heating bath* at the instant that the sample becomes *transparent.*

Time Requirements:

Sample Preparation.—Powdering and packing samples into melting point tubes and attaching tubes to thermometer—about 10 min.

Test Procedure.—Immersing melting point tube assembly in heating bath; raising bath to within 15 C. of softening point and then 1 C. per min.—25 to 35 min.

Reproducibility:

Theoretical.—No statement made.

Actual.—About 5 C., especially between different operators.

Reference:

"Manual of Analytical Methods," R60-4b Hercules Powder Co., October 30, 1939.

XIX. HERCULES THERMOMETER DROP METHOD FOR DETERMINING THE SOFTENING POINT OF ROSIN

Principle of Method:

Based on the characteristic gravitational flow of sample from the bulb of a thermometer under specified temperature conditions.

Bath System:

Consists of an air sample bath within a liquid heating bath using water, glycerine, or other suitable liquid as a constant temperature heating medium.

Apparatus:

Thermometer having a bulb $\frac{1}{4}$ in. (6.5 mm.) in diameter and $\frac{5}{8}$ in. (16 mm.) in length.

Sample Bath Container.—Test tube $\frac{7}{8}$ in. (22 mm.) in diameter and 7 to 8 in. (178 to 203 mm.) in length.

Heating Bath Container.—800 to 1000-ml. beaker.

Sample Preparation:

About 25 g. of sample melted at 100 to 120 C. and 0.50 to 0.55 g. molded on warmed thermometer bulb by dipping and rotating in melted sample.

Cooled at room temperature for about 15 min.

Thermometer, supported by cork, suspended in test tube.

Test Procedure:

Heating bath adjusted to 9 to 14 C. above the expected softening point.

Test tube containing thermometer with sample, immersed in the *heating bath* with *temperature* maintained *constant* throughout test.

Bath Agitation:

Heating bath agitated.

Softening Point (Meaning of Term):

Temperature of the air sample bath and sample at the instant that, flowing from the thermometer bulb, the elongated drop first becomes constricted to form a tear.

Time Requirements:

Sample Preparation.—Melting and molding on thermometer bulb and cooling for 15 min. to room temperature—20 to 25 min.

Test Procedure.—Raising temperature from room temperature to softening point—5 to 10 min.

Number of tests that can be made simultaneously—2 or more.

Reproducibility:

Theoretical.—No statement made.
Actual.—1.0 to 1.5 C.

Reference:

Softening Point (Thermometer Drop Method) from "Manual of Analytical Methods—1944 Revision;" Hercules Powder Co.

XX. GENERAL ELECTRIC THERMOMETER DROP METHOD FOR DETERMINING THE MELTING POINT OF BITUMINOUS MATERIALS

Principle of Method:

Same as for Method XIX.

Bath System:

Consists of an air sample bath within a liquid heating bath using sulfuric acid as a heating medium.

Apparatus:

Thermometer.—No specification given.
Sample Bath Container.—Test tube $\frac{5}{8}$ in. (16 mm.) in diameter and 6 in. (152 mm.) in length with side tubulature.
Heating Bath Container.—600-ml. beaker.

Sample Preparation:

Sample softened and 1 g. molded on the bulb of a thermometer.

Cooled to room temperature and thermometer, supported by cork, suspended in test tube.

Test Procedure:

Test tube containing thermometer with sample immersed in heating bath.

Rate of raising the temperature of the air sample bath not given.

Bath Agitation:

No agitation.

Softening (or Melting) Point (Meaning of Term):

Temperature of the air sample bath and sample at the instant that the softening sample, flowing from the thermometer bulb, elongates sufficiently to touch the bottom of the test tube, a distance of $\frac{3}{4}$ in. (19 mm.).

Time Requirements:

Sample Preparation.—Softening and molding on thermometer bulb and cooling to room temperature—estimated 20 to 25 min.

Test Procedure.—Raising temperature of air sample bath to melting point—(unknown to author).

Number of tests that can be made simultaneously with same heating bath—1 or more.

Reproducibility:

Theoretical.—No statement made.
Actual.—Unknown to author.

Reference:

Roy Cross, "Handbook of Petroleum, Asphalt and Natural Gas."

XXI. DRIP METHOD FOR THE DETERMINATION OF THE MELTING POINT OF ROSIN

Principle of Method:

Same as for method XIX.

Bath System:

Consists of an air sample bath within a liquid heating bath using water as a heating medium.

Apparatus:

Thermometer.—Having a bulb 6 to 7 mm. ($\frac{1}{4}$ in.) in diameter and 9 to 13 mm. ($\frac{3}{8}$ to $\frac{1}{2}$ in.) in length.
Sample Bath Container.—Test tube, 22 mm. ($\frac{7}{8}$ in.) in diameter and 200 mm. (8 in.) in length.
Heating Bath Container.—Low-form or ordinary 600-ml. beaker.

Sample Preparation:

A quantity of sample (about 10 ml.) is

melted in a 18-mm. diameter test tube by immersing the tube in boiling water.

A 0.5-g. portion is molded on warm thermometer bulb by dipping in the melted sample.

Cooled to room temperature and allowed to remain at this temperature for 10 min.

Thermometer, supported by cork, suspended in test tube so that end of bulb is 25 mm. (1 in.) above bottom of tube.

Test Procedure:

Test tube containing sample on thermometer bulb, immersed in heating bath adjusted to a temperature of not over 45 C.

Temperature of the sample bath (and sample) raised at the rate of not more than 2 C. per min. up to 65 C. and at a rate of 1 C. per min. above 65 C.

Bath Agitation:

No agitation.

Softening (or Melting) Point (Meaning of Term):

Temperature of the air sample bath and sample at the instant that, flowing from the thermometer bulb, the sample elongates sufficiently to touch the bottom of the test tube, a distance of 25 mm. (1 in.).

Time Requirements:

Sample Preparation.—Melting sample, molding on thermometer, and allowing to stand for 10 min. after cooling—20 to 25 min.

Test Procedure.—Raising temperature at rate of 2 C. to 65 C. and then at rate of 1 C.—25 to 50 min.

Number of tests that can be made simultaneously with one heating bath—1 or more.

Reproducibility:

Theoretical.—If variation of four determinations is more than 1 C., an additional determination shall be made.

Actual.—Greatest variation by different laboratories in 1926 referee tests by A.S.T.M. Committee D-17—36 C.

References:

Description of method as sent out to collaborating laboratories in 1926 by A.S.T.M. Committee D-17 for referee melting point tests.

Description of method in "Physical and Chemical Examination—Paints, Varnishes, Lacquers, Colors," Gardner, Sixth Edition, pp. 721–722 (1933).

XXII. A.S.T.M. CUBE-IN-WATER METHOD (D 61 - 38) FOR PITCHES HAVING SOFTENING POINTS BETWEEN 43 AND 80 C.

Principle of Method:

Based on the characteristic gravitational flow of a sample from a wire support under specified temperature conditions.

Bath System:

Consists of a liquid sample bath serving also as a heating bath using water as a heating medium.

Apparatus:

Cubical Mold 12.7 mm. (½ in.) inside.

L-Shape Wire Hook made of No. 12 B. & S. gage copper wire with foot 25.4 mm. (1 in.) in length.

Heating Bath Container.—600-ml. beaker, Griffin low-form or equivalent.

Sample Preparation:

Sample melted and poured or softened and pressed into mold.

Cooled at room temperature.

Removed from mold and placed on hook with wire piercing center of cube.

Test Procedure:

Sample cube and thermometer immersed in the water bath at a temperature of 15.5 C. (60 F.) for 15 min.

Temperature of the bath raised at the uniform rate of 5 C. (9 F.) per min.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of the water bath at the instant the softening sample elongates sufficiently to touch the bottom of the beaker, a distance of 25.4 mm. (1.0 in.).

Time Requirements:

Sample Preparation.—Melting and pouring into mold, cooling and placing on hook—estimated 20 to 30 min.

Test Procedure.—Holding at temperature of 15.5 C. for 15 min. and raising at rate of 5 C. to softening point—estimated 20 to 30 min.

Number of tests that can be made simultaneously with one bath—1.

Reproducibility:

Theoretical.—Results should not differ from each other by more than 0.5 C. (1 F.).

Actual.—Unknown to author.

Reference:

A.S.T.M. Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (D 61 – 38), 1946 Book of A.S.T.M. Standards, Part II, p. 608.

XXIII. JOHNSON AND JOHNSON CUBE-IN-AIR METHOD FOR ROSIN AND PITCH

Principle of Method:

Same as for Method XXII.

Bath System:

Consists of an air sample bath within a liquid heating bath using water as a heating medium.

Apparatus:

Brass Cubical Mold ½ in. (13 mm.).

L-Shape Wire Hook made of No. 12 B. & S. gage with ⅞ in. (22 mm.) foot, vertical length 5 in. (127 mm.).

Sample Bath Container.—Suitable size test tube provided with cork having required openings.

Heating Bath Container.—1500-ml. Griffin low-form beaker.

Sample Preparation:

Sample melted and poured into mold and foot of hook inserted in center of cube while hot.

When cold, cube removed from mold and placed in refrigerator overnight.

Test Procedure:

Cube suspended with thermometer in air sample bath so that the cube is 1½ in. (38 mm.) from the bottom of the test tube.

Test tube with sample immersed in water heating bath at 20 C. for 5 min.

Heating bath temperature raised at uniform rate of 5 C. per 3 min. (3 F. per min.).

Bath Agitation:

Heating bath agitated.

Softening Point (Meaning of Term):

Temperature of air sample bath at the instant that the softening sample elongates to within ½ in. (13 mm.) of the bottom of the test tube, a distance of 1 in. (25 mm.).

Time Requirements:

Sample Preparation.—Melting and pouring sample in mold, inserting hook, and cooling—estimated 20 to 30 min. plus overnight.

Test Procedure.—Holding at 20 C. for 5 min., then raising at rate of 3 F. per min. to softening point—estimated 20 to 45 min.

Number of tests that can be made simultaneously with one bath—1.

Reproducibility:

Theoretical.—Limit of accuracy is plus or minus 0.5 C. (or 1 F.).

Actual.—Unknown to author.

Reference:

Description and drawing sent by W. C. Nicholas to J. M. Schantz and acknowledged by C. E. Kinney, September 29, 1932.

XXIV. N.Y.T.L. METHOD FOR THE DETERMINATION OF THE MELTING POINT OF BITUMINOUS SUBSTANCES

Principle of Method:

Based on the characteristic gravitational flow of sample out of and from a metal mold or container into air under specified temperature conditions.

Bath System:

Consists of an air sample bath within a liquid heating bath using cottonseed oil as a heating medium.

Apparatus:

Sample Bath Container.—200-ml. tall-form lipless beaker.

Heating Bath container.—600-ml. Griffin low-form beaker.

Sample Holder Assembly consisting of two heavy parallel brass plates about $2\frac{3}{8}$ in. (60 mm.) apart joined by adjustable posts, four sample holders, and thermometer. The upper plate of assembly, serving as cover for the sample bath container, is 3 in. in diameter and $\frac{1}{4}$ in. in thickness and has central opening for thermometer cork. The lower plate for holding sample molds is 2 in. in diameter and $\frac{3}{8}$ in. in thickness and has four openings threaded and central opening $\frac{3}{8}$ in. in diameter for thermometer.

Sample Molds are A.S.T.M. standard float collars, 9.5 mm. ($\frac{3}{8}$ in.) in diameter at small end, 12.7 mm. ($\frac{1}{2}$ in.) at large end, and 22.2 mm. ($\frac{7}{8}$ in.) in length, threaded for screwing into lower plate.

Preparation of Sample:

About 20 g. of sample melted; portion poured into mold and cooled in air to room temperature.

With mold screwed into place, assembly suspended in water bath at 25 C. (77 F.) for not less than 15 min.

Test Procedure:

Sample holder assembly transferred from water conditioning bath to air sample bath at 25 C. (77 F.) with mold holder adjusted so that it is $1\frac{1}{4}$ in. (3.2 mm.) from bottom of beaker.

Temperature of air sample bath raised at uniform rate of 5 F. (4 C.) per min.

Bath Agitation:

No agitation.

Melting Point (Meaning of Term):

Temperature of air sample bath at instant softening sample, flowing out of and from the mold, elongates sufficiently to touch the bottom of the beaker, a distance of $1\frac{1}{4}$ in.

Time Requirements:

Sample Preparation.—Melting, pouring into mold, cooling, and holding sample at 25 C. for at least 15 min.—estimated about 30 min.

Test Procedure.—Transferring holder assembly to air sample bath and raising temperature to melting point at rate of 5 F. per min.—estimated 5 to 15 min.

Number of tests that can be made simultaneously with one heating bath—1 or more.

Reproducibility:

Theoretical.—Duplicate tests should agree within plus or minus 2 F.

Actual.—Unknown to author.

Reference:

Description and drawing of assembly by the Barber Asphalt Company (1935). Furnished C. E. Kinney, 1943.

XXV. DENNIS MELTING POINT APPARATUS—ELECTRICALLY HEATED BAR METHOD

Principle of Method:

Based on the physical change, at the softening point, of a powdered sample distributed over the surface of an electrically heated bar having a heat gradient between terminals adjusted to a temperature range which includes the softening point.

Bath System:

No bath system.

Apparatus:

Metal Bar heated electrically so that a heat gradient of about 100 C. exists along the bar. A movable arm is provided with a constantan needle which makes contact with any point throughout its length and which in turn is connected with a potentiometer connected to the left end of the bar.

Sample Preparation:

Sample powdered.

Test Procedure:

Sample sprinkled lightly on *heated bar*.

Bath Agitation:

No agitation.

Softening Point (Meaning of Term):

Temperature of the bar at the point above which the powdered sample is molten and below which it is still solid.

Time Requirements:

Sample Preparation.—About 5 min.
Test Procedure.—1 min.

Reproducibility:

Theoretical.—Not stated.
Actual.—Not better than 2 or 3 C. on resins.

References:

L. M. Dennis and R. S. Shelton, "An Apparatus for the Determination of Melting Points," *Journal*, Am. Chem. Soc., Vol. 52, Part 2, pp. 3128–3130 August, 1930.

"British Standard Specifications for Synthetic Resins," British Standards Institution, No. 474 (1932).

XXVI. CONSTANT-TEMPERATURE AIR BATH MODIFICATION OF THE A.S.T.M. BALL AND RING METHOD FOR SOFTENING POINT

Principle of Method:

Same as for A.S.T.M. Method E 28–42 T.[2]

Apparatus and Preparation of Sample:

Sample melted and poured into brass ring, as in Method E 28. A single ring in special ring holder is inserted into stoppered air bath chamber of restricted volume, immersed in liquid bath. Bulb of air bath thermometer held in fixed position close to sample.

Test Procedure:

Temperature of air bath is controlled and dependent on temperature of liquid bath, which is held constant at a point, selected on basis of preliminary test or knowledge of sample, that is between 5 and 15 C. above the expected or observed softening point reading. Advantage lies in ability to remove tested sample and holder from air bath and immediately reintroduce holder with fresh sample for test.

Softening Point (Meaning of Term):

Air-bath thermometer reading at end of test is used uncorrected when it differs from constant liquid bath temperature by from 8 to 10.5 C. If difference is greater or less, but within the 5 to 15 C. limit stated above, the observed air-bath temperature reading must be corrected by means of an empirical formula.

Reproducibility:

Determinations made at varying bath temperatures within the limits stated, when corrected as indicated, had maximum deviations from the mean value of less than 2 C. Duplicate determinations made at any one bath temperature usually agreed within less than 0.5 C. and never varied by more than 1 C.

Time Requirement:

Approximately 10 min. per single test, without loss of time between tests.

Reference:

Grotlisch and Burstein, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 17, August 15, 1945, p. 476.

REPORT OF COMMITTEE D-18*
ON
SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes held two meetings during the year: one at Atlantic City, N. J., on June 17, 1947, and one at Washington, D. C., on March 2, 1948. The Executive Committee met at Washington, D. C., on November 30, 1947.

The reorganization of the committee was approved by the committee members and became effective after the June, 1947, meeting. The basis of the new organization is the grouping of subcommittees into three groups, namely, Administrative, Research, and Standards. Most of the subcommittees in the first and second groups have completed their organization. Chairmen of the new Administrative and Research Subcommittees were appointed. Only one Standards Subcommittee has been established at the present time to receive proposed standards from all Research Subcommittees with the second vice-chairman appointed as the chairman. Two minor changes were made in the By-laws: (*1*) The secretary was authorized to appoint an assistant secretary instead of the personnel secretary originally proposed, and (*2*) the By-laws require two meetings of the committee to be held each year, one of which was designated as the annual meeting. F. J. Converse and W. G. Holtz were appointed second vice-chairman and assistant secretary, respectively.

A Symposium on Load Testing and three papers of general interest were presented at the 1947 Annual Meeting. The symposium is now published as a special compilation and the three papers have been published in the 1947 *Proceedings*.[1]

The special Subcommittee on Procedures in Soil Testing, under the chairmanship of M. D. Catton, was charged with the responsibility of revising the A.S.T.M. Manual on "Procedures for Testing Soils." The original manual was reprinted during the year, without revisions, to supply the large demand for it. During the next few months the various state highway testing laboratories and soils engineers and laboratories of the various government services such as U. S. Engineers, U. S. Navy, Civil Aeronautics Administration, etc., will be contacted to learn of test methods now in use and which should be included in the book. At the same time suggestions will be requested for other test methods that should be included. It is anticipated that some little time will be required to assemble the information desired.

Several new consultant members were appointed to the various subcommittees during the year, these members being chosen in order to obtain more information with respect to soils problems in the specific field of the subcommittee to which they were appointed.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 408 (1947).

The chairman and several members will be absent from the committee meetings during the 1948 Annual Meeting because of attendance at the Second International Soil Conference at Rotterdam, Holland. E. J. Kilcawley and F. J. Converse were appointed to represent the Society and Committee D-18 at the Conference. At the meeting in June the following two papers of general interest will be presented: "Importance of Relative Density in Studying the Behavior and Characteristics of Soils,"[2] by D. M. Burmister and "The Determination of Limits for the Control of Placement Moisture in High Rolled Earth Dams,"[3] by W. G. Holtz.

The committee is making a survey to determine the probable attendance at a proposed meeting of the committee to be held in conjunction with a meeting of the Society in San Francisco, Calif., in October, 1949.

At the March meeting of the committee, the present officers and Executive Committee members were reelected for the ensuing term of two years.

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status without revision:

Tentative Method of:
Test for Moisture-Density Relations of Soils (D 698 - 42 T),
Test for Specific Gravity of Soils (D 854 - 45 T),
Mechanical Analysis of Soils (D 422 - 39 T), and
Tentative Definition of:
Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee G-2 on Special Papers (C. W. Allen, chairman).—No special papers were solicited this year because of the activities of members in preparing papers for the Second International Soil Conference. Two papers of general interest will be presented at the main committee meeting in June.

Subcommittee G-3 on Nomenclature and Definitions (E. A. Abdun-Nur, chairman).—Liaison with the corresponding Committee on Glossary of Terms and Definitions of the Soil Mechanics and Foundation Division, of the American Society of Civil Engineers, has been established. The subcommittee plans to work on the revision of the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) during the coming year.

Subcommittee G-4 on Evaluation of Data (E. A. Willis, chairman) discussed a report entitled "A Revised Method of Interpretation of Triaxial Compression Tests for the Determination of Shearing Strength," which had been submitted to the chairman by Mr. Douglas McHenry.

It was decided to circulate the report to the membership for their review and recommendation regarding publication. It was also decided to refer the report to the chairmen of the Research Subcommittees as illustrating the type of report Subcommittee G-4 was interested in. Finally, it was decided that the report should be called to the attention of Subcommittee R-5C since that subcommittee is concerned with methods of testing soils under Triaxial loading.

Subcommittee R-5, Section A, on Consolidation (D. M. Burmister, chairman) has initiated a program of research for this year in order to obtain basic information for setting up reasonable and practical requirements and procedures for a consolidation test. This program includes: The influence of type of loading, in respect to both uniform increment loading *versus* loading in increments with a ratio of 2 to 1, as usually done, and increment *versus* continuous loading; influence of the time interval between applications of successive increments of load; and direct permeability test method of determination of the coefficient of consolidation *versus* the analytical methods now commonly used.

---

[2] See p. 1249.
[3] See p. 1240.

In addition, the section contemplates going over the nomenclature and symbols which apply to consolidation, in the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) with a view of revising them, if necessary, and of adding such new terms as may be considered advisable.

Subcommittee R-5, Section B, on Direct Shear (F. J. Converse, chairman) has under development a tentative method for making direct shear tests. This method has not yet reached a stage where it can be presented as tentative.

Subcommittee R-5, Section C, on Methods of Testing Soils under Triaxial Loading (W. G. Holtz, chairman) met in June, 1947, to discuss the various methods of performing and interpreting trixial compression tests. One paper of general interest was presented under the auspices of the subcommittee. The subcommittee will hold a meeting in June, to discuss and review two reports on recent triaxial compression developments.

Subcommittee R-6 on Physicochemical Properties of Soil (E. F. Preece, chairman).—Generally, the fine-grained soils problem consists, at least at present, of two distinct lines of investigation: first, studies concerning the significance of the various physicochemical properties and, second, the methods of identification and measurement of those individual properties.

Although unorganized as yet, the principal function of Subcommittee R-6 will be, first, the development of unified procedures. Even this very preliminary step may not be feasible at first and unification may of necessity have to start with unification of elements of a procedure. The second step will be the development of tentative methods. This cannot be undertaken, however, until it has been established that the property under consideration is a significant one with respect to behavior and the nature of its measurement well established. The third step, of course, is standardization of the procedure.

It appears advisable to approach the problem of physico-chemical test procedures as individual units as, for example, X-ray diffraction, differential thermal analysis, exchange capacity, and exchangeable bases, etc.

Because of the tremendous amount of work that must be completed in order to make the application of X-ray diffraction methods to soil identification practicable, it is planned to consider this procedure first.

Subcommittee R-7 on Identification and Classification of Soils (G. W. McAlpin, chairman).—The membership of the committee provides representation of all major soil engineering divisions, as well as specialists on subjects of geology, pedology, identification of soils from air photos, clay mineralogy, and soil chemistry.

At the meeting of the subcommittee on March 1, 1948, in Washington, D. C., it was agreed that the committee should: (*1*) Stress the use of the word "identification" and minimize the term "classification" when speaking of committee activities; (*2*) formulate a definition of the soil components; (*3*) develop a "minimum" identification of individual soil samples, and that this "minimum" identification should be supplemented by the engineer and other specialists. The degree of identification required would in all cases depend upon that needed for the analysis of the engineering problems at hand.

Subcommittee R-9 on Dynamic Properties of Soils (R. K. Bernhard, chairman) may be considered, to a certain extent, a continuation of the former sub-committee on Soil Dynamics with an enlarged membership.

The following are considered as possible fields within the subcommittee's sphere of interest: (*1*) Theoretical problems on the dynamic constants, dynamic stability, resonance phenomena, wave study, etc., and (*2*) engineering problems, such as, compaction, bearing capacity depths effected by vibration, vibration shielding, traffic vibrations, etc. A preliminary canvass of research activities in the field of soil dynamics has been made.

Subcommittee R-10, Section A, on Load Bearing (L. A. Palmer, chairman).—An outline of major items considered as included in the scope of the work of this subcommittee has been prepared and discussed.

Subcommittee R-10, Section B, on Pile Bearing (A. E. Cummings, chairman) held a meeting at Washington on March 2, 1948. The subcommittee decided to attempt to develop a tentative method for load tests on piles. This would cover only the test procedure with no effort being made to include a discussion of the interpretation of the test results.

This report has been submitted to letter ballot of the committee, which consists of 82 voting members; 60 members returned their ballots, of whom 56 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. Kilcawley,
Chairman.

R. F. Blanks,
Secretary.

REPORT OF COMMITTEE D-19*

ON

INDUSTRIAL WATER

Committee D-19 on Industrial Water and its subcommittees held regular meetings at Atlantic City, N. J., June 19 and 20, 1947, and at Washington, D. C., March 1 and 2, 1948.

The committee is sponsoring jointly with the Joint Research Committee on Boiler Feedwater Studies a Session on Water to be presented at the June, 1948, Annual Meeting of the Society at Detroit, Mich. The session is in the form of a panel discussion on "The Influence of Non-Ferrous Metals and Their Compounds on the Corrosion of Pressure Vessels." It includes the following papers:[1]

Station Design and Composition of Materials as Factors in Boiler Corrosion, by R. B. Donworth, Duquesne Light Co.

Corrosion of High Pressure Steam Generators: The Status of Our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes, by R. C. Corey, Bureau of Mines.

Numerous policy matters studied by the officers of the committee during the year, were presented and approved at the March 2, 1948 meeting of the committee. These are as follows:

(*a*) Change in name of the committee to "Committee D-19 on Industrial Water."

(*b*) Expansion in committee activities with the organization of a new Subcommittee VII on Water-Borne Industrial Wastes.

(*c*) Revisions in by-laws of the committee.

(*d*) Revision in general scope of the committee and preparation of scope for the new Subcommittee VII.

(*e*) Discontinuance of the designation "Recommended Practice" by issuing all methods as "Methods".

Effective arrangements have been concluded between the A.S.T.M. and The American Society of Mechanical Engineers in setting up a liaison group including the chairman and present members of Section C, Sampling of Steam, of Subcommittee III, Methods of Sampling, and members from Committee No. 19 on Instruments and Apparatus of the A.S.M.E. Power Test Code Committee. To secure wider attention to this work, the "Proposed Method for the Sampling of Steam" has been published in the A.S.T.M. BULLETIN.[1a]

Substantial progress was made during the year on the proposed A.S.T.M. Manual on Industrial Water. Topical outlines of each of the chapters have been prepared and approved by letter ballot of the committee members. Manuscripts of the first draft of three of the chapters are being studied.

Chairman Max Hecht, the Society's representative on the Executive Committee of the Joint Research Committee on Boiler Feedwater Studies, attended two meetings at Atlantic City, one on June 18, the other on December 3, 1947. F. N. Speller was represented by E. P. Partridge at the December 3 meeting. W. C. Schroeder, was elected the Research Committee Chairman, S. F.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] See pp. 897 and 907.

[1a] ASTM BULLETIN, No. 151, March, 1948, p. 34.

Whirl and R. W. Seniff, Vice-Chairmen and T. H. Daugherty, Secretary. Arrangements have been made for presentation in 1948 of programs sponsored by the Research Committee at various national Society meetings. The Detroit program, mentioned above, is one of the co-sponsored sessions.

Chairman Max Hecht, the Society's representative on the Feedwater Subcommittee of the A.S.M.E. Boiler Code Committee, attended two meetings, one of the working group at Pittsburgh, Pa., November 12, 1947, the other of the Feedwater Subcommittee at Atlantic City, N. J., December 4, 1947. E. P. Partridge, chairman of the working group, presented suggestions secured from members of the Feedwater Subcommittee to the third draft, "Suggested Care of Stationary Steam Boilers, A.S.M.E. Boiler Construction Code, Section VII-B." The working group's report was reviewed at the December 4 meeting of the Feedwater Subcommittee, C. W. Rice, chairman. The revised draft dated December 4, 1947, was referred to the proper group of the Boiler Code Committee with the recommendation that it be published in the appropriate A.S.M.E. periodical for comments.[2]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman,	Max Hecht.
Vice-Chairmen,	L. K. Herndon
	F. R. Owens
Secretary,	R. E. Hall.

NEW TENTATIVES

The committee recommends for publication as tentative the following two methods as appended hereto:[2a]

Tentative Method of Test for Nitrate Ion in Industrial Waters.—This is the first of a series of methods for determining nitrogenous substances in industrial water.

Tentative Method of Test for Sulfate-Reducing Bacteria in Industrial Waters and Water-Formed Deposits.—This is the second of a series of methods using the biological microscope for the detection and identification of biological organisms that form deposits on surfaces in contact with industrial waters.

Both of these methods have been submitted for review to Committee E-1 on Methods of Testing.

REVISIONS OF TENTATIVES

Tentative Recommended Practice for Field Sampling of Water-Formed Deposits (**D 887 - 46 T**).[3]—Committee D-19 recommends that this Recommended Practice be changed to a Method of Test with revisions in the text as appended hereto.[4]

Tentative Method of Test for Total Aluminum and Aluminum Ion in Industrial Waters (**D 857 - 45 T**).[3]—Committee D-19 recommends the following revisions in this method:

Section 2 (a).—Change the second sentence to read as follows: "In addition, it is possible to distinguish aluminum ion and colloidal aluminum from suspended aluminum by determining total aluminum and ionic aluminum, the difference between the two being suspended aluminum."

Section 4.—Add the following sentence: "See Note 1 on purity of hematoxylin in Section 5 (*i*)."

Section 5 (h).—Change to read as follows: "(*h*) *Glycerine Solution.*—Dilute 1 volume of glycerine with 1 volume of distilled water."

Section 5 (i).—Designate the present note as "Note 2" and add the following as "Note 1."

---

[2] *Mechanical Engineering*, June, 1948.

[2a] These methods were accepted as tentative by the Society and appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

[3] 1946 Book of A.S.T.M. Standards, Part III-A.

[4] This revised method was accepted by the Society and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-A.

NOTE 1: *Caution.*—Hematoxylin to be suitable for this determination should be of a tan and not red color. When in doubt of the quality of the hematoxylin reagent, the following test should be performed. Take a sample of water containing approximately 0.25 ppm. of aluminum as Al and proceed in accordance with Section 6 (*f*) and (*g*). If the hematoxylin is satisfactory, a blue color will develop.

Section 6 (*a*).—Change the first sentence to read as follows: "Immediately after shaking the sample, measure accurately into a 100-ml. Erlenmeyer flask, a volume of sample that will contain from 0.001 to 0.005 mg. of aluminum."

Section 6 (*f*).—Change the first sentence to read as follows: "Add 10 ml. of glycerine solution and 5 ml. of hematoxylin solution to each flask, mix and allow to stand for 15 min."

Section 7.—Change to read as follows: "Follow the procedure described in Section 6, except that a filtered or decanted portion of the sample, free from suspended material, shall be used."

Delete the present note in this section and substitute the following: "Note.—The aluminum ion and colloidal aluminum are both determined by this procedure."

Tentative Method of Field Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (**D 807 - 46 T**).[3]—Committee D-19 recommends the following revision of this method:

Section 12 (*b*).—Replace the present paragraph and note with the following paragraph and notes:

(*b*) A boiler water may be considered to have had no embrittling tendencies during the period of the tests when successive 30-, 60-, and 90-day tests each have shown no cracking of the test specimens.

NOTE 1.—Since the character of a boiler water may change with variation in the water supply or the chemical treatment of the water any conclusions based upon the condition of the test specimens are strictly valid only with respect to the actual periods of exposure.

NOTE 2.—Inasmuch as a personal element is involved in the operation of the detector, there is an uncertainty factor in a test that results in no cracking. Correlation of more than 500 tests made by average plant personnel indicates that the probable chance of error is no more than 5 per cent after 30-, 60-, and 90-day tests have been completed. The Society will welcome reports of additional tests for future correlation.

Fig. 3.—Change "15°" to read "14°."

Fig. 4.—Insert 5/16 in. as the horizontal dimension of the bevelled 10° edge of the clamping plate.

Tentative Recommended Practice for Corrosion Tests in Industrial Waters (*NDHA Method*) (**D 935 - 47 T**).[5]—The committee recommends that "recommended practice" be replaced by "method" in the title and in the text wherever it occurs.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (**D 513 - 43**).[3]—The committee recommends the complete revision and reversion to tentative of this method as appended hereto,[4] which includes the substitution of new apparatus based on investigation at the U. S. Navy Engineering Experimental Station at Annapolis, Md. by Frank E. Clarke.[6]

Recommended Practice for Sampling Boiler Water from Stationary Boilers (**D 860 - 47**).[5]—The committee recommends that "recommended practice" be changed to read "method" in the title and text, and that this method be reverted to tentative.

The committee also recommends the following revision of this method:

Section 1.—Add "for special investigations" to the end of the first sentence in Paragraph (*a*). Delete Paragraph (*b*) and reletter Paragraph (*c*) as (*b*).

---

[5] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[6] "Determination of Carbon Dioxide in Water," by F. E. Clarke, *Analytical Chemistry*, Vol. 19, p. 889 (November, 1947).

Methods Continued Without Revision

The committee recommends that the following methods be continued in their present status as they are currently being evaluated and may require revision:

Standard Methods for Determination of:[3]

Calcium Ion and Magnesium Ion in Industrial Waters (D 511 – 42),
Chloride Ion in Industrial Waters (D 512 – 42),
Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 – 43),
Sulfate Ion in Industrial Waters (D 516 – 42), and

Tentative Methods of Test for:[3]

Dissolved Oxygen in Industrial Waters (D 888 – 46 T.)

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

Activities of Subcommittees

Subcommittee I, Advisory (Max Hecht, chairman) adopted the following policies which were approved by members present at the March 2, 1948, meeting of Committee D-19, and have been referred to the committee membership for letter ballot action.

(*1*) Revision of By-Laws and scopes of the committee and subcommittees shown in The Regulations Governing A.S.T.M. Committee D-19 on Industrial Water. The major revisions include change in the name of the committee to read: "Committee D-19 on Industrial Water"; provisions to permit the inclusion of two vice-chairmen in place of one; editorial revision to the general scope of the committee; and the inclusion of the scope of the new Subcommittee VII on Water-Borne Industrial Wastes. The revised regulations are subject to letter ballot approval by the committee.

(*2*) Establishment of a new Subcommittee VII on Water-Borne Industrial Wastes.

(*3*) Final arrangement by the Society officers, at the request of Committee D-19, for setting up of a liaison group between the A.S.M.E. and A.S.T.M. to forward the cooperative work in the establishment of Methods for the Sampling of Steam, as referred to earlier in this report.

(*4*) Issuance of all methods as tentative or standard methods in place of designating them as recommended practices, inasmuch as Committee D-19 is concerned almost exclusively with macorial that would be subject to tentractural agreement. Tentative and standard methods of test and testing shall include procedures bearing the name "referee" and, when suitable procedures are found, "non-referee," and that a note appear following the title "non-referee" to indicate that it is included because of its usefulness as a rapid control method of analysis. The designation "Proposed Method" shall be applied from the inception of any method, and retained throughout both subcommittee and committee discussions. The scope and general direction of a proposed method after it has been developed by the subsection shall be submitted to the Advisory Subcommittee for information, suggestion, and approval. The Advisory Subcommittee shall decide on the designation to be recommended to the Society, its decision shall be confirmed by vote of the committee. Proposed methods, when released either by the subcommittee or committee, shall be published "for information" as an Appendix to the Annual Report and in the Manual when there is any indecision that the proposed method cannot be accepted as tentative.

---

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

The present recommended practices now published in the 1946 Book of A.S.T.M. Standards and in the 1947 Supplement will be referred back to the appropriate subcommittee for review and recommendation for disposition.

Subcommittee II, Editorial (E. P. Partridge, chairman) approved all of the new methods and revisions recommended by Committee D-19 in this report. As noted earlier it reports progress by Section A on the preparation of the A.S.T.M. Manual on Industrial Water. In addition, it has prepared suggestions to serve as a guide for the subcommittees in the preparation of the form of proposed methods, such preparation to be made at the source, namely, the subsection. These guides have been issued in the minutes of the committee.

Subcommittee III on Methods of Sampling (F. R. Owens, chairman).—Section A on Sampling of Water is studying revisions for the Tentative Methods of Sampling Water for Industrial Uses (D 510 – 47 T) particularly for the sampling of water containing unstable constituents and dissolved gases. Progress has been reported on a Proposed Method for Sampling of Water under Subatmospheric Pressure and on Specifications for Sampling Equipment.

Section B on Water-Formed Deposits prepared the revision of D 887 previously mentioned.

Section C on Sampling of Steam is cooperating with the A.S.M.E. on methods for sampling of steam, as has been referred to earlier in this report.

Subcommittee IV on Methods of Analysis (A. A. Berk, chairman) submitted a number of revisions which are referred to earlier in this report.

Section A on Analytical Methods has been supplied with copies of a paper, "The Determination of Dissolved Oxygen in Deaerated Boiler Feedwater," by J. R. McDermet, presented at the Water Conference at Pittsburgh, Pa., November 13, 1947. The data in this paper supplements that presented at the 1947 Water Sessions. This section is currently studying revisions in Methods D 511, D 512, D 515, D 516, and D 888 previously mentioned. It also originated the revisions in Methods D 513 and D 857 and the new method for nitrate.

The subsections are working on the initial drafts for proposed methods which include alkali metals, sodium and potassium, iron, residual chlorine, hardness, and nitrites. The Subsection on Carbon Dioxide has in preparation nonreferee procedures. The Subsection on Dissolved Gases is developing methods for ammonia and hydrogen sulfide. The Subsection on Silica is developing new procedures for this determination. Subsections on Acidity and Alkalinity and on Dissolved Solids intend to offer reports at an early date.

Section B on Electrometric Methods expects to submit at an early date a draft of a proposed method of test for electric conductance of industrial waters. This section is also working on the preparation of directions for the application of the Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T) in industrial waters. This section plans to cooperate with Section C on Special Methods in preparing methods for similar evaluation of instruments for colorimetric analysis.

Section C on Special Methods prepared the proposed Tentative Method of Test for Sulfate-Reducing Bacteria in Industrial Waters, previously mentioned. It also prepared the Proposed Method for Identification of Types of Microorganisms in Industrial Waters,[8] published as information only as appended hereto. As a result of the new policy regarding the discontinuance of recommended practices, this section is revising the Tentative Recommended Practice for Application of X-Ray Diffraction Methods to Water-Formed Deposits (D 934 – 47 T).[5] The section has in preparation methods for the detection of algae and also for the inspection of water-formed deposits covering the application of the petrographic microscope, the flame photometer, and the spectroscope.

---

[8] See p. 486.

Subcommittee V on Classification (L. K. Herndon, chairman) intends to present at the Annual Meeting typical analyses, both surface and ground supplies, as a key for both water treatment and interpretation of troubles encountered in industry.

Subcommittee VI on Methods of Testing (V. V. Kendall, chairman) prepared the revisions of Methods D 807 and D 935 referred to earlier in this report. Subsections are currently being organized to undertake the work of preparation of specifications for synthetic sea water and methods for conducting service tests of pipes.

This report has been submitted to letter ballot of the committee, which consists of 84 members; 66½ members returned their ballots, of whom 56½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

APPENDIX

PROPOSED METHOD FOR IDENTIFICATION OF TYPES OF MICRO-ORGANISMS IN INDUSTRIAL WATERS[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method covers the identification by microscopic examination of bacterial cells, mold filaments, and such particles of inert material as may be found in industrial water samples.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Centrifuge.*—A two-tube centrifuge with a variable-speed motor is adequate.

(*b*) *Centrifuge Tubes.*—Conical type centrifuge tubes are preferable.

(*c*) *Cover Glasses.*—Round or square type cover glasses, $\frac{3}{4}$ in. in diameter, may be used. These shall be perfectly clean and grease free.

(*d*) *Immersion Oil.*—Cedar wood or Crown oil is suggested.

(*e*) *Light Source.*—A 75- to 100-w., nitrogen-filled, tungsten bulb with a "daylight" glass filter is satisfactory.

(*f*) *Microscope.*—A microscope that will provide magnifications of from 400 to 1000 diameters. Par-focal lenses and a mechanical stage are convenient but not necessary.

(*g*) *Pipets.*—10-ml. Mohr-type pipets with a 3- to 4-mm. opening, and 1-ml. Mohr type pipets may be used. The wide-mouth 10-ml. pipet makes the transfer of thick sediment from the sample bottle more convenient.

(*h*) *Slides.*—Standard 1 by 3-in. slides with either plain or frosted ends are preferable. These shall be perfectly clean and grease free.

NOTE.—A satisfactory method for cleaning slides is as follows:[3] Place the slides in glacial acetic acid for 15 min.; then wash in distilled water. Place in 95 per cent alcohol for 5 min. and dry with a clean towel. Pass through a bunsen flame before using.

Reagents

3. (*a*) *Ammonium Oxalate - Crystal Violet Solution.*—Prepare Hucker's modification of the Gram stain[4] as follows:

(*1*) Prepare Solution A:

Crystal violet (90 per cent dye content)	2.0 g.
Ethyl alcohol (95 per cent)	20 ml.

(*2*) Prepare Solution B:

Ammonium oxalate	0.8 g.
Distilled water	80 ml.

(*3*) Mix Solutions A and B.

(*b*) *Counterstain for Gram Stain.*[4]—Prepare as follows:

Safranin O (2.5 per cent solution in 95 per cent alcohol)	10 ml.
Distilled water	100 ml.

(*c*) *Dorner's Nigrosin Solution.*[4]—Prepare as follows:

(*1*) Prepare an aqueous Nigrosin solution as follows:

Nigrosin (water-soluble). (American nigrosins certified by the Commission on Standardization of Biological stains are satisfactory)	10 g.
Distilled water	100 ml.

---

[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-19 on Industrial Water.

[2] Published as information, June, 1948.

[3] J. A. Kolmer and F. Boerner, "Approved Laboratory Technique," Second Edition, Appleton-Century Publications, p. 56 (1938).

[4] "Manual for Pure Culture Study of Bacteria," Biotech Publications, Geneva, N. Y., Leaflet IV$_{46}$, pp. 6 to 8 (1946).

TABLE I.—KEY FOR IDENTIFICATION OF MICROORGANISMS.

I. FILAMENTOUS STRUCTURES:

A. *Algae.*—These filaments are, in general, larger than either mold or bacterial filaments and usually contain a greenish pigment (chlorophyll) which is lacking in molds or bacteria. The use of the low-power objective of the microscope is generally sufficient to distinguish these types.[5]

B. *Molds.*—These structures are, in most cases, much smaller in diameter than the algae and the high-power objective of the microscope is generally used for identification. They lack pigments and are usually larger in diameter than bacterial filaments. Mold filaments may be differentiated from other structures, such as vegetable fibers, that are likely to be found in water samples by observing the following points:[6]

(*1*) *Segmentation.*—Most mold filaments appear segmented by the occurrence of cross walls, and the segments are generally several times longer than the width of the filament. *Phycomycetes* are an exception with no cross walls.

(*2*) *Granulation.*—Under higher magnifications, fine granules are visible within the mold filaments.

(*3*) *Parallelism of the Sides of the Cell Wall.*—The two side walls of mold filaments are always parallel. In other structures, the sides of the filament may appear sharply constricted or expanded.

(*4*) *Comparison of Focal Planes of Side Walls.*—The two side walls of the mold filament are always in focus at the same level, while the side walls of other filaments may come into focus at two different levels. This results from the fact that mold filaments are tubular or cylindrical, while other structures may be ribbon-like.

(*5*) *Character of Branchings.*—Among mold filaments, branching occurs at abrupt angles, while in other structures branching may be rather indefinite, the branches curving gently away from the main body of the filament.

(*6*) *Appearance of Ends of Filaments.*—The ends of mold filaments are blunt, never flared or pointed.

C. *Bacteria.*—Bacterial filaments differ from mold filaments chiefly in their smaller size. The use of the oil-immersion objective is necessary for their identification. Many of the filamentous bacteria belong to the family *Chlamydobacteriales* and these are described in detail in the Tentative Method for Detection of Iron Bacteria in Industrial Waters (A.S.T.M. Designation: D 932).[7] There is another type of filamentous bacterium belonging to the family *Beggiatoaceae*[8] commonly found in sewage-polluted waters. Their filaments sometimes attain a length of from 50 to 100 microns and may be from 2 to 6 microns in diameter. They exhibit a slow, waving motion like that of the alga, *Oscillatoria*, and contain numerous oil-like droplets of free sulfur. Another common type that forms filaments is the spore-forming group. The filaments are from 0.5 to 2 microns in diameter and may be as long as 50 to 100 microns. They have definite cross walls and some dense areas in addition to the spore.

II. NONFILAMENTOUS STRUCTURES:

A. *True Bacteria.*—These may be distinguished, in wet mounts, from other nonfilamentous particles by their relatively uniform size and shape. True bacteria occur as either rods, spheres (cocci), or spiral forms which vary from a comma-shape to a true spiral. Some bacteria are motile and this motility, when present, is observed to be a definite forward or backward motion; this tends to distinguish it from the slight oscillatory motion due to the Brownian movement of all finely divided particles. Bacterial cells may be more definitely differentiated from crystalline or amorphous particles which are likely to be included in water samples by employing one of the various staining techniques, as follows:

(*1*) *Negative Stainings.*—This method does not stain the bacteria but instead stains the background black. The bacterial cells then appear as light areas in a darkened field. However, only the outlines of the organisms are made apparent by this method. A method for negative staining is as follows:[4] Place a small drop of the sediment on a glass slide, mix with an equal volume of Dorner's Nigrosin solution (Section 3(*c*)), and streak out with the narrow edge of another slide. The streak or film resulting should be thin, since thick smears are likely to crack and peel upon drying. Dry the slide in air and examine under the oil-immersion lens without a cover glass.

(*2*) *Staining with Aniline Dyes.*—Aniline dyes are employed whenever it is desirable to obtain, in addition to the outline of the bacterial cells, some indication as to their internal structure. Before employing the aniline dyes, place a small drop of the specimen on a glass slide and spread out in a thin film. Allow the film to air-dry and fix it by passing it through a Bunsen flame several times. Care shall be taken not to overheat the slide as this tends to cause distortion.

---

[5] A detailed description of algae will be included in the Proposed Method for Identification of Algae, which is in preparation by Committee D-19.

[6] F. W. Tanner, "The Microbiology of Foods," Second Edition, Garrard Press, Champaign, Ill., p. 678 (1944).

[7] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[8] Martin Frobisher, Jr., "Fundamentals of Bacteriology," Third Edition, W. D. Saunders Co., p. 398 (1945).

TABLE I.—KEY FOR IDENTIFICATION OF MICROORGANISMS—(*Continued*).

(*a*) A simple method for staining with an aniline dye[4] employs Loeffler's methylene blue solution (Section 3(*e*)). Flood the dye over the smear and allow to remain in contact with it for about 1 min. Wash off the dye with a gentle stream of cool water and blot the slide dry between two pieces of blotting paper. Examine the slide under the oil-immersion lens. The bacterial cells will be stained blue; spores, if present, will not take the stain; and granular structures will be a darker color than the cell.

(*b*) Gram's differential stain offers a more detailed method, not only for definition of the cell structure, but also for classification of the organisms. The method is as follows:[4] Apply the ammonium oxalate - crystal violet solution (Section 3(*a*)) for 60 sec., wash gently with water, and apply the iodine solution (Section 3(*f*)) for 60 sec. Wash this off in turn. Apply ethyl alcohol (95 per cent) and renew until all but the thickest portion of the smear ceases to give off the purple dye (usually about 20 to 30 sec.). Wash with water to stop the decolorization, and flood the smear with the counterstain (Section 3(*b*)), which shall be left on the slide for about 20 to 30 sec. Then blot the smear dry and examine under the oil-immersion lens. In this technique, the application of the alcohol is the important step and the one whereby the differentiation as to Gram-positive or Gram-negative is made. Those bacteria which will retain the violet - iodine combination are referred to as Gram-positive and exhibit a dark purple color when examined microscopically. Those which are decolorized by the alcohol and subsequently take up the counterstain are referred to as Gram-negative. Safranin used as a counterstain gives these a light red color. The Gram's stain is meant to be used for bacteria that are 24 hr. old or less. As the bacteria become older, Gram-positive organisms frequently lose this characteristic and appear Gram-negative. The presence of Gram-variable organisms may mean that the bacteria are of different ages or that different types are present.

B. Algae.—Unicellular types of algae may be observed which carry the characteristic green pigment within the cells. These may occur singly or in groups and are frequently enclosed in a gelatinous material. The diatoms are recognized by the definite shape of their siliceous shells and by the characteristic markings on them. Living diatoms are indicated by the presence of chlorophyll consisting of green or brown pigment in the form of droplets within the cell while dead diatoms may be entirely empty. Protozoa may be observed which may or may not contain green colored materials. They generally have a definite internal structure and move quite rapidly.[5]

C. Crystalline Material.—Unlike bacteria, crystalline particles found in a water sample are likely to vary considerably in size from relatively tiny grains to a size easily apparent to the naked eye. They exhibit definite outlines which are generally angular and may be seen to reflect light from several different planes. They may or may not possess color.

D. Amorphous Material.—The amorphous material found in a water sample appears as aggregates of finely divided particles without any definite shape.

(*2*) Immerse in boiling water bath for 30 min.; then add as a preservative:

Formalin	0.5 ml.

(*3*) Filter through double filter paper and store in sterile serological test tubes, about 5 ml. to the tube.

(*d*) *Ethyl Alcohol* (*95 per cent*).

(*e*) *Loeffler's Methylene Blue* (*Emended Statement*).[4]—Prepare as follows:

(*1*) Prepare Solution A:

Methylene blue (90 per cent dye content)	0.3 g.
Ethyl alcohol (95 per cent)	30 ml.

(*2*) Prepare Solution B:

KOH (0.1 g. per l.)	100 ml.

(*3*) Mix Solutions A and B.

(*f*) *Lugol's Iodine Solution.*—Prepare Gram's modification of Lugol's solution[4] as follows:

Iodine	1.0 g.
Potassium iodide	2.0 g.
Distilled water	300 m.

Procedure

4. (*a*) In examining water samples microscopically, first describe the physical appearance of the sample. This description should indicate the color of the sample, its turbidity, and the relative amount, color, and consistency of any sediment or flocculent material contained therein.

(*b*) With samples containing a visible sediment, the specimen for microscopic examination may be obtained by use of a pipet. With the forefinger held over the mouthpiece of the pipet, insert the tip into the sample bottle immediately over the sediment. Release the finger slightly, allowing a small amount of the sediment to be drawn into the pipet.

Place a small drop of the sediment so obtained upon a glass slide.

(*c*) If the sample contains no visible sediment, obtain sufficient material for microscopic examination by centrifuging for 30 min. at a moderate speed. After centrifuging, decant the supernatant liquid to within $\frac{1}{4}$ in. of the tip of the tube. In case the material remaining in the tube is not sufficiently turbid, repeat the process by pouring an additional portion of the sample into the tube, centrifuging again, and again decanting the supernatant liquid. Place a drop of the turbid fraction on a glass slide by means of a glass rod or by tilting the centrifuge tube and allowing the material from the tip to pour down the side of the tube.

(*d*) Place a cover glass on the drop of material on the slide, care being taken to avoid air bubbles under the cover glass. This may be done by tilting the glass at an angle of about 45 deg., with the lower edge of the glass touching the material on the slide. Carefully lower the glass until it rests flat over the specimen. Crush or flatten thick particles by gently tapping the cover glass, since thinner mounts make for clearer detail under the microscope. Remove any excess moisture with filter paper or a towel.

(*e*) Bring the specimen into focus with the low-power objective of the microscope, examine briefly, and then observe more closely with the "high-dry" objective. In examining wet mounts prepared as described in Paragraph (*d*), it will be found that contrast between such hyaline (colorless) structures as bacterial cells and the water in which they are suspended is poor in the presence of excessive illumination. This can be remedied by partly closing the iris diaphragm of the substage condenser, lowering the condenser, and/or readjusting the angle of the substage mirror.

(*f*) Structures may be found with the "high-dry" objective that warrant greater magnification, and for this purpose the oil-immersion lens shall be used. Swinging the "high-dry" lens out of the way, place a drop of immersion oil on the cover glass over the area illuminated by the light entering from the substage condenser. Swing the oil-immersion objective into place. Watching the lowest point of this objective *from the side*, *carefully* lower it with the coarse adjustment until it is immersed in the oil and nearly touching the cover glass. Then, looking through the eyepiece, bring the specimen into focus by raising or lowering the objective with the fine adjustment. The amount of light entering upon the specimen may need adjusting at this point.

(*g*) In examining stained smears, the same general technique shall be used in focusing with the oil-immersion lens. However, in this case, maximum illumination is desired. Open the iris diaphragm completely, focus the substage condenser, and adjust the mirror so as to reflect light from the brightest part of the light source.

Identification or Differentiation

5. Structures likely to be found in water samples may be classified in accordance with the key shown in Table I.

Evaluation and Report

6. A report of a microscopic examination of the material contained in a water sample shall indicate in a proportional manner the occurrence of the structures noted: many, occasional, or few. When the specimen is found to be composed of a mixture such as bacteria, molds, crystalline, and amorphous material, their relative predominance shall be stated. In cases where the sample has been concentrated by means of a centrifuge, this fact shall be stated; otherwise the report might be misleading.

REPORT OF COMMITTEE D-20*

ON

PLASTICS

Since the June, 1947, meeting at Atlantic City, N. J. Committee D-20 on Plastics and its subcommittees have held meetings in Atlantic City, N. J., in November, 1947, and at Dayton, Ohio, in February, 1948. Meetings were also held at these times by the Advisory Committee which also held a special meeting at A.S.T.M. Headquarters in Philadelphia, Pa., in January, 1948.

Since the Secretary, Mr. A. J. Warner, left for England, Committee D-20 appointed Mr. B. L. Lewis to fulfill his unexpired term.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, G. M. Kline.
First Vice-Chairman, L. W. A. Meyer.
Second Vice-Chairman, R. K. Witt.
Secretary, B. L. Lewis.
Membership Secretary, T. J. Dietz.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative:

Method of Test for Weight Loss of Plastics on Heating (D 948 – 47 T), Recommended Practice for Molding Specimens of Phenolic Materials for Use in Electrical Tests (D 949 – 47 T).

Revision of Tentative Specifications for:

Cellulose Acetate Molding Compounds (D 706 – 46 T), and
Cellulose Acetate Butyrate Molding Compounds (D 707 – 46 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, as well as in the 1948 Compilation of A.S.T.M. Standards on Plastics.

On March 18, 1948, the Standards Committee accepted the following recommendations:

New Tentative Methods of:

Test for Apparent Density and Bulk Factor of Non-Pouring Molding Powders (D 954 – 48 T).

Revision of Tentative Methods of:

Test for Measuring the Molding Index of Thermosetting Molding Powder (D 731 – 44 T), and
Test for Specific Gravity of Plastics (D 792 – 44 T).

Withdrawal of Tentative Specifications for:

Non-Rigid Ethyl Cellulose Plastics (D 743 – 44 T).

The new and revised tentatives will appear in the 1948 Compilation of A.S.T.M. Standards on Plastics.

The following recommendations were accepted by the Standards Committee on June 7, 1948:

New Tentative Method of:

Measuring Shrinkage From Mold Dimensions of Molded Plastics (D 955 – 48 T).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

New Tentative Recommended Practice for:

Molding Specimens of Amino Plastics (D 956 - 48 T).
Determining Mold Surface Temperature of Commercial Molds for Plastics (D 957 - 48 T), and
Determining Temperatures of Standard A.S.T.M. Molds for Test Specimens of Plastics (D 958 - 48 T).

Revision of Tentative Methods of:

Test for Deformation of Plastics Under Load (D 612 - 45 T).

The new and revised tentatives will appear in the 1948 Compilation of A.S.T.M. Standards on Plastics.

Recommendations Affecting Standards

The committee is recommending the adoption as standard of eight tentatives and revisions in seven tentatives. The tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

Activities of Subcommittees

All of the subcommittees have been quite active during the year. The work of the subcommittees on specifications, analytical procedures and definitions has expanded. The Subcommittee on Research is still attracting a large audience at its meetings.

Subcommittee I on Strength Properties (M. E. Marks, chairman).—The work is still continuing on tensile and compressive strength in relation to tensile strength of films, the effect of speed, and the strength of rods and tubes. A new falling-ball test is being evaluated so that a method can be written up in the near future. Various methods of evaluating plastics in fatigue as well as shear are likewise under study. The new bearing strength method has been completed[2] but work in this field is still continuing. Bonding strength of laminates is now available but work is continuing along this line of laminates made from other plastics not yet tested.

Subcommittee II on Hardness (R. B. Rice, chairman) has been quite active during the year and has completed a revision of the Rockwell hardness test method (D 785 - 44 T). Other hardness methods are under study and work is being done on the Rockwell method using the alpha scale.

During the year it was found advisable to make two sections of the Abrasion Resistance Section, one for determining the optical effects while the new section, which was established this year, is working on the problem of abrasion resistance for mechanical effects.

Subcommittee III on Thermal Properties (E. B. Cooper, chairman) has been active in the field of flammability and flow of thermal plastic materials. It has completed tests for measuring molding index of thermosetting molding powder. Revisions to the Method of Measuring Deformation of Plastics Under Load (D 621 - 45 T) was completed.

A flammability test for thin plastic film is under consideration and the study of stiffness for laminates at elevated temperature is under development.

During the year it was found necessary to establish a new section for setting up a method for measuring the properties of a laminate known as "postformability."

Subcommittee IV on Optical Properties. (G. W. Ingle, chairman) has in process a considerable amount of work.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[2] See Editorial Note, p. 493.

At the present time there is under consideration a simple method of determining light transmission. The study of haze is being continued, in order to derive equations for properly computing haze by the general electric spectrophotometer or the integrating sphere hazemeter.

A new section was organized recently for determining optical properties requiring the goniophotometric measurement for gloss and diffusion.

A new section was set up very recently to study the question of luminous reflectance and transmission.

The Section on Optical Distortion was disbanded since further work was not considered advisable. Significance of test statements are being prepared for a number of the present methods.

Subcommittee V on Permanence Properties (J. H. Teeple, chairman).—Further study is being made of the test for colorfastness of plastics to light (D 620 – 45 T) as regards the distance from the lamp to the specimen. A method for measuring water vapor permeability at 100 F. is under development. Work is also to be undertaken on the development of a gas permeability test. Other matters being studied include tests for shrinkage, exudation and crocking.

Subcommittee VI on Specifications (F. W. Reinhart, chairman).—During the year the various sections have been working on specifications for molding compounds and also plasticizers. Revisions in several of the specifications are being recommended this year.

Work on polyethylene as well as cellulose acetate propionate and cellulose propionate is going ahead and specifications are being prepared.

Subcommittee VII on Analytical Methods (P. D. Bross, chairman) has under consideration methods for the analysis of plasticizers in cooperation with Subcommittee VI. It is now engaged in methods for studying acidity, ester content, specific gravity, and color of plasticizers.

Subcommittee VIII on Research (J. D. Ryan, chairman) is continuing its work on the fundamental aspects of impact and flow properties of plastics. Work has been initiated on the degradation of plastic materials.

A list of research problems suitable for investigation by graduate students in educational institutions has been prepared and is published in the A.S.T.M. BULLETIN, January, 1948. Papers of an original nature are requested and solicited for future meetings of this subcommittee.

Subcommittee IX on Molds and Molding (L. A. Sontag, chairman).—Work in subcommittees has continued on molding procedures for thermosetting molding compounds. The methods for measuring mold shrinkage, and for determining temperature of standard A.S.T.M. test specimen molds, also the recommended practice for molding specimens of amino plastics, as well as the method for determining mold surface temperatures of commercial molds were prepared by this subcommittee.

A round-robin test has been completed on transfer dies for test specimens and a method will be written up. This is also true of the use of tension specimens for impact tests. There is also under consideration, the preparation of a method for molding 2-in. diameter disks of phenolic materials.

Subcommittee X on Definitions, Nomenclature, and Significance of Test (G. W. Clark, chairman) has been working on many significance of tests statements. Work is being continued on definitions of additional terms. The subcommittee is giving further consideration to changes in the definitions for film and sheeting.

The code for direction of testing specimens will shortly be submitted to ballot of Committee D-20.

This report has been submitted to letter ballot of the committee which consists of 157 voting members; 71 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

ROBERT BURNS,
Chairman.

B. L. LEWIS,
Secretary.

EDITORIAL NOTE

On June 23, 1948, the Administrative Committee on Standards accepted the recommendation of Committee D-20 that the Method of Test for Bearing Strength of Plastics be approved for publication as tentative. The new tentative method appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B, bearing the designation D 953 - 48 T.

The proposed Tentative Method of Test for Bond Strength of Plastics and Electrical Insulating Materials was accepted as tentative by the Standards Committee on July 16, 1948. The new tentative method has been assigned the designation D 952 - 48 T, and appears in the 1948 Supplement to Book of A.S.T.M. Standards, Part III-B.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are given proposed revisions in certain tentatives covering plastics, which are referred to earlier in this report. These tentatives appear in their present form in the 1946 Book of A.S.T.M. Standards, Part III-B or its 1947 Supplement to Part III-B, as indicated by the final number in the A.S.T.M. designation.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following five tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision.

Tentative Methods of Test for:

- Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (D 758 - 44 T),
- Tensile and Compressive Properties of Plastics at Subnormal and Supernormal Temperatures (D 759 - 44 T),
- Long Time Tension Tests of Plastics (D 674 - 46 T),
- Tensile Strength of Molded Electrical Insulating Materials (D 651 - 42 T), and
- Deviation of Line of Sight Through Transparent Plastics (D 881 - 46 T).

The committee recommends that the Tentative Methods of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 - 44 T) and Designating the Flow Temperature of Thermoplastic Molding Materials (D 863 - 45 T) be combined and approved for reference to letter ballot of the Society for adoption as standard with the addition of the following statement on significance of test:

> 2. *Significance of Test.*—The equilibrium state of the material in this test is not necessarily representative of the state at which the material is molded commercially. The indicated temperature of the heat-transfer medium in an injunction or extrusion machine cylinder always is much in excess of that of the material itself and of the flow temperature of the material. Thus, the chief use of this test is for control and identification purposes rather than for prediction of molding conditions or physical properties.

The committee recommends that the Tentative Method of Test for Estimating Blocking of Plastic Sheets (D 884 - 46 T) be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

Section 1.—Change to read as follows:

> 1. This method is intended to give information regarding the degree of blocking of plastic sheets under specified conditions of temperature, humidity, and pressure.

Section 2.—Replace the present Section 2 by a new section on significance of test, to read as follows:

> 2. *Significance of Test.*—Blocking is the adhesion between touching layers of a material, such as occurs under moderate pressures during storage or use. When deciding on the degree of blocking to be permitted, the investigator should consider the conditions under which the plastic sheet is stored, the manner in which it is processed, and its ultimate use. For example: Plastic transparent sheets used for handwrapping of candy boxes are shipped in the form of cut-to-size sheets, stacked about 5 in. high in each container. They are stored indoors at moderate temperatures. In the packing operation the sheets are taken one by one from the stack, and in this operation the sheets should slide freely. Suitable conditions of testing would therefore be a pressure of $\frac{1}{3}$ psi. and a temperature of 50 C.,

and the permissible degree of blocking would be Degree N.B. (no blocking). In contrast, a sheet to be used for raincoats may be stored in use under high pressure while wet, but a slight blocking will not be objectionable as long as the surface is not damaged when separating the layers. Such a material might be tested under a pressure of 1 psi., at 38 C., in a saturated atmosphere, and the permissible degree of blocking can be Degree Sl. B. (slight blocking). Similar considerations should govern the selection of testing procedure and selection of permissible degree of blocking in other applications.

TABLE I.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Grade 5	Grade 6	Grade 8
Specific gravity, 25/25 C. (77/77 F.), max.	1.19	1.19	1.19
Index of refraction, 25 C. (77 F.) (approximate, for clear material)			
Min.	1.48	1.48	1.48
Max.	1.50	1.50	1.50
Luminous transmission of 1/8-in. clear material, min., per cent	90	90	90
Water absorption, 24-hr. immersion, max., per cent:			
Weight gain plus soluble matter loss	0.5	0.5	0.5
Soluble matter loss	0.1	0.1	0.1
Heat distortion temperature, min.:			
At 264 psi. fiber stress			
deg. Cent.	65	72	88
deg. Fahr.	149	162	190
At 66 psi. fiber stress			
deg. Cent.	68	76	95
deg. Fahr.	154	169	203
Tensile strength, min., psi.:			
At 25 C. (77 F.)	8 000	8 000	9 000
At 70 C. (158 F.)	2 000	3 000	4 000
Flexural strength at 25 C. (77 F.), min., psi.	14 000	14 500	15 000
Impact strength (Izod), min., ft. lb. per in. of notch:			
At 25 C. (77 F.)	0.4	0.4	0.4
At −40 C. (−40 F.)	0.3	0.3	0.3
Flammability (for 1/8- to 1/4-in. thicknesses) max., in. per min.	1.8	1.8	1.8
Dielectric constant (for unfilled material), max.:			
At 60 cycles per second	4.5	4.5	4.5
At 1,000 cycles per second	4.0	4.0	4.0
At 1,000,000 cycles per second	3.5	3.5	3.5
Dissipation factor (for unfilled material), max.:			
At 60 cycles per second	0.05	0.05	0.06
At 1,000 cycles per second	0.04	0.04	0.05
At 1,000,000 cycles per second	0.03	0.03	0.03
Deformation under load at 2000 psi., max., per cent at 50 C. (122 F.)	4.0	3.0	2.0
Flow temperature			
deg. Cent. ±5 C.	135	140	155
deg. Fahr. ±9 F.	275	284	311

REVISIONS OF TENTATIVES

Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (D 747 – 43 T):

Title and Scope.—Delete "nonrigid" from both title and scope.

Section 5.—In the fourth sentence of Paragraph (*a*), change "shortest span" to read "longest span."

At the end of this section, add the following note:

NOTE.—An effort shall be made to utilize as much as possible of the range of both the load scale and the angular deflection scale by judicious choice of specimen width and thickness, moment weights, and span, during any single test.

Tentative Specifications for Laminated Thermosetting Materials (D 709 – 47 T):

Committee D-20 joins Committee D-9 on Electrical Insulating Materials in recommending revision of these specifications. This revision consists of the addition of four glass-base grades of materials together with the specified requirements and tolerances. The revision is given in full in the Appendix to the Report of Committee D-9.[1]

Tentative Specifications for Methacrylate Molding Compounds (D 788 – 44 T):

Section 2.—Change to read as follows:

2. *Grades.*—These specifications cover the following three grades of polymethyl methacrylate base molding compounds:

Grade	Heat Distortion Temperature at 264 psi., min.	
	deg. Cent.	deg. Fahr.
5	65	149
6	72	162
8	88	190

---

[1] See p. 413.

Section 3.—Change the note under this section to read as follows:

NOTE.—Clear, colored, or pigmented molding compounds suitable for injection, compression, or extrusion molding are usually supplied in granulations that will pass a No. 3 (¼-in.) sieve or a No. 6 (3360-micron) sieve.

Clear powders, suitable for compression molding, are usually supplied in a pearl form having a particle size distribution such that none will remain on a No. 10 (2000-micron) sieve and 20 to 75 per cent will pass a No. 60 (3360-micron) sieve.

Table I.—Change to read as shown in the accompanying Table I.

Tentative Specifications for Polystyrene Molding Compounds (D 703 - 44 T):

Sections 2, 3, and Table I.—Delete all references to Type 3.

Section 5 (j).—Add the following sentence: "The test for deformation under load shall be made at a load of 4000 psi. and a temperature of 50 C."

Change the requirements for dielectric constant for type 1 at all frequencies from a range of 2.50 to 2.75 to 2.45 to 2.65.

Change the requirements for dielectric constant for type 2 at all frequencies from a range of 2.50 to 2.60 to 2.45 to 2.60.

Tentative Specifications for Cellulose Acetate Compounds (D 706 - 47 T):

It is proposed to delete three grades (Type I Grade H4, Type II MH, and Type IV Grade MS) from the specifications and to assign reference numbers to the grades remaining. It is also proposed to change the temperature for high temperature tensile strength from 71 C. (160 F.) to 70 C. (158 F.) to conform to standard A.S.T.M. test temperatures.

Section 2 and Table I.—Change to read as follows:

2. These specifications cover four types and 13 grades of cellulose acetate molding compounds, classified in accordance with their flow temperatures as shown in Table I (the accompanying Table II).

Table II.—Delete the requirements for type I, grade H4; type II, grade MH; and type IV, grade MS.

Under the grade designations in the title of Table II, add the corresponding formula reference numbers as given in Table I above.

In the first column of Table II, change "Tensile strength at 71 C. (160 F.)" to read "Tensile strength at 70 C. (158 F.)."

Section 6 (e).—In the last sentence in this section, change "71 C. (160 F.)" to read "70 C. (158 F.)."

TABLE II.—TYPES AND GRADES OF CELLULOSE ACETATE MOLDING COMPOUNDS.

Type	Grade	Formula Reference Number	Flow Temperature	
			deg. Cent.	deg. Fahr.
I. General Purpose...	H2	1	160 ±5	320 ±9
	MH	2	150 ±5	302 ±9
	MS	3	140 ±5	284 ±9
	S2	4	130 ±5	266 ±9
II. Heat Resistant....	H6	5	180 ±5	356 ±9
	H4	6	170 ±5	338 ±9
	H2	7	160 ±5	320 ±9
III. Impact Resistant..	MH	8	150 ±5	302 ±9
	MS	9	140 ±5	284 ±9
	S2	10	130 ±5	266 ±9
IV. Moisture Resistant	H4	11	170 ±5	338 ±9
	H2	12	160 ±5	320 ±9
	MH	13	150 ±5	302 ±9

Tentative Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 44 T):

Scope.—Delete Paragraphs (*b*) and (*c*).

New Section.—Add a new section on significance of test to read as follows, renumbering the subsequent sections accordingly:

2. *Significance of Test.*—(*a*) The results obtained by this method are a measure of the indentation produced by the penetrator described in the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18) when applied as described in the Method E 18 and in Sections 5 and 6 of this Method D 785. Method A measures the indentation remaining 15 sec. after a given major load has been released from the penetrator after

being applied for a given length of time. Method B measures the indentation resulting from a given load being applied by the penetrator for a given period of time; the measurement being made with the major load applied.

(*b*) For certain types of materials having creep and recovery, the time factors involved in applications of major and minor loads have a considerable effect on the results of the measurements.

(*c*) The results obtained by this method are not generally considered a measure of the abrasion or wear resistance of the plastics.

Section 5.—Renumber as Section 6, and change the title to read "Method A."

New Section.—Add the following Method B as a new Section 7, to read as follows:

7. *Method B.*—(*a*) The procedure described in Sections 2 to 7 of A.S.T.M. Methods E 18 shall be followed.

(*b*) All tests shall be made at a temperature of 25 ± 2 C. (77 ± 3.6 F.), unless otherwise specified.

(*c*) For this test the ½-in. penetrator and the 60-kg. major load shall be used.

(*d*) The "spring constant" or correction factor of the machine shall be determined as follows: A soft copper block shall be placed on the anvil in the normal testing position. Using the ½-in. penetrator, the sample shall be raised to the normal starting position with the minor load applied and the dial set to zero. The major load then shall be applied by tripping the lever. The dial gage will then indicate the vertical distance of indentation plus the spring of the frame and any other elastic compressive deformation of the penetrator spindle and penetrator. This operation shall be repeated several times without moving the block, but resetting the dial to zero if necessary before applying the major load, until the deflection of the dial gage becomes constant; that is, until no further indentation takes place and only the spring of the instrument remains. This value, in terms of dial divisions, is the correction factor which shall be used in Paragraph (*f*).

(*e*) Following the machine adjustment described in Section 6 (*d*), the test specimen shall be placed in position on the anvil. With the specimen in place, the minor load of 10 kg. shall be applied and the zero setting made within 10 sec.; the major load shall be applied ***immediately*** after the zero setting has been completed. The total number of divisions that the needle of the dial gage passes through during 15 sec. of major load application shall be observed. It is to be noted that the numbers shown on the standard dial gage are not to be used, but that the actual scale divisions representing indentation shall be counted. A full revolution of the needle represents 100 divisions. It is also to be noted that the limitations given in Section 2 still apply.

(*f*) The total scale divisions that are indicated by the dial gage, as observed in accordance with Paragraph (*e*), represent the indentation produced ***plus*** the spring constant correction of the test instrument. This correction factor shall be subtracted from the observed reading, and the difference recorded as the total indentation under load.

(*g*) *Calculation.*—The hardness by this method, to be known as the alpha (α) Rockwell hardness number, shall be calculated as follows:

Alpha Rockwell hardness number = 150 − total indentation under load

NOTE: *Examples.*—With a total indentation of 30 divisions, obtained as described in Paragraphs (*e*) and (*f*), the α value is α 120; or if the total indentation is 210 the α value is $\alpha - 60$.

Section 6.—Change to read as follows:

6. The report shall include the following:

(*1*) The method used (Method A or B),

(*2*) For Method A, the Rockwell hardness number read on the red scale. The letter R, L, M, or E shall precede the Rockwell hardness number, depending on the combination that has been used in determining the hardness,

(*3*) For Method B, the alpha Rockwell hardness number preceded by the symbol α,

(*4*) The conditioning procedure used,

(*5*) The total thickness of the specimen, and

(*6*) The number of pieces in the specimen and their average thickness.

Tentative Recommended Practice for Molding Specimens of Phenolic Materials (D 796 - 45 T):

Table I.—Change the requirements for molding temperature for specimens of shock-high resistant phenolic materials from "155 ± 3 C." to read "150 ± 3 C."

REPORT OF COMMITTEE E-1*

ON

METHODS OF TESTING

Committee E-1 on Methods of Testing held one meeting during the year in Washington, D. C., on March 4, 1948, in connection with the spring group meetings of A.S.T.M. committees.

Since its reorganization last year the committee has been giving consideration to expediting the review of methods and requirements for apparatus prepared by other A.S.T.M. technical committees. It appreciates that the decision to discontinue the group of representative members will make it all the more necessary for the individual sections and technical committees to be fully representative from the viewpoint of the interested technical committees of the Society. This particular change in the committee structure will receive further consideration during the coming year.

The committee has in preparation a revised set of Regulations Governing Committee E-1 in line with the changes made in its reorganization.

Committee E-1 will have several recommendations affecting standards to present to the Society this year. These actions are discussed later in this report under the activities of the particular technical committees or sections in which they originated. These several recommendations will be presented to the Administrative Committee on Standards subsequent to the Annual Meeting.

The Board of Directors of the Society has appointed Messrs. L. H. Fry, D. E. Parsons, T. S. Taylor, and R. L. Templin each for another term of three years as members of Committee E-1.

The committee accepted with regret the resignation of Professor H. F. Moore as chairman of the Advisory Committee of Technical Committee I on Mechanical Testing, and also as chairman of the Section on Calibration of Testing Machines and Apparatus. Mr. B. L. Wilson of the National Bureau of Standards has been appointed chairman of the Section on Calibration.

Professor F. B. Seely resigned as chairman of the Section on Elastic Strength of Materials and Mr. F. M. Howell of the Aluminum Company of America has been appointed chairman of this section.

Subsequent to the 1947 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:

Verification of Calibration Devices for Verifying Testing Machines (E 74 - 47 T).

Revision of Tentative Specifications for:

A.S.T.M. Thermometers (E 1 - 46 T).

Revision and Reversion to Tentative of:

Standard Methods of Verification of Testing Machines (E 4 - 36).

These recommendations were accepted by the Standards Committee on November 6, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards.

On February 16, 1948, the Standards Committee accepted revisions of the Tentative Methods of Impact Testing of Metallic Materials (E 23 - 41 T). The revised tentative appears in the 1947 Supplement to Book of A.S.T.M. Standards, Parts I-A and I-B.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing:

Meetings of five of the sections of this technical committee were held in Washington, D. C., during the week of March 1, 1948, at the time of the spring group committee meetings. The work carried on during the year is covered in the following brief summary of activities.

Section on Tension Testing (R. L. Templin, chairman).—This section has been very active in reviewing new methods prepared by other Society committees. This has included methods for cleavage strength of metal to metal adhesives, test for strength properties of adhesives in shear by tension loading, tension test methods for copper bars and shapes, tension test for steel spring wire, tension test for thin copper and copper alloy sheet and strip, and for fine wire and ribbon used in electronic devices and lamps. A number of comments and suggestions resulting from this review have been submitted to the technical committees that prepared these methods.

The section has expressed the hope that it would not be necessary to have special tension testing methods for each type of metallic material considered by the Society. In the case of these particular methods it was considered possible to make changes in the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46) that would take care of the points required in the testing of copper bars and shapes, thin copper sheet, and steel spring wire.

Section on Compression Testing (M. F. Sayre, chairman).—At a meeting held in Atlantic City, N. J., on June 16, 1947, consideration was given to the second draft of proposed Tentative Methods of Compression Testing of Metallic Materials in Sheet Form. It is hoped that it may be possible to complete these methods during the year so that they can be recommended for publication as tentative.

A proposed Tentative Method of Test for Compressive Strength for Magnesium Oxychloride Cement, prepared by Committee C-2 on Magnesium Oxychloride Cements, was reviewed by members of the section and a number of comments and suggestions submitted to the originating committee.

Section on Impact Testing (W. W. Werring, chairman).—Revision of the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 47 T) was completed during the year. At a meeting in Washington, D. C., on March 2, 1947, the section gave further consideration to the information in these methods on the significance of tension-impact tests. It was decided to revise and condense this information. The revised information on significance, when completed, will be placed in an appendix to the methods. The section is also compiling information on impact test specimens now specified in test codes and standards of other organizations.

Section on Flexure Testing (H. F. Clemmer, chairman).—This section held a meeting in Washington, D. C., on March 3, 1948, at which consideration was given to a program of work. The section has under review a proposed Tentative Method of Test for Transverse Strength of Magnesium Oxychloride Cement Compositions Using Simple Bar with Knife Edge or Two Point Loading, prepared by Committee C-2.

Section on Effect of Speed of Testing (P. G. McVetty, chairman).—This section is sponsoring the Symposium on Speed of Testing[1] to be held at this

---

[1] See pp. 1129 to 1200.

Annual Meeting of the Society, at which the following five papers will be presented:

"Testing Speed Limitations for Committee A-1 Specifications for Steel," by Lawford H. Fry.
"The Effect of Speed of Testing on Magnesium-Base Alloys," by A. A. Moore.
"Speed of Testing Wood: Factors in Its Control and Its Effect on Strength," by L. J. Markwardt and J. A. Liska.
"Effect of Speed of Test Upon Strength Properties of Plastics," by Albert G. H. Dietz, W. J. Gailus, and S. Yurenka.
"Methods and Equipment for Controlling Speed of Testing," by Lawrence K. Hyde.

The Bibliography on Effect of Speed of Testing on the Results of Physical Tests of Various Materials, which contains some 265 references to articles published from 1906 to 1946, was completed during the year and copies are obtainable from A.S.T.M. Headquarters.

This section is giving consideration to a study of speed requirements for nonmetallic materials and to a correlation of speed requirements in various A.S.T.M. specifications.

Section on Elastic Strength of Materials (F. M. Howell, chairman).—A meeting of this section under its new chairman was held in Washington, D. C., on March 3, 1948, at which plans were made for a program of work to be undertaken. This will include a review of definitions of terms relating to methods of testing prepared by other Society committees. In this connection the section is now reviewing a series of definitions appearing in several methods of testing plastics prepared by Committee D-20 on Plastics.

Technical Committee II on Consistency, Plasticity, and Related Properties (W. F. Fair, Jr., chairman):

Section on Softening Point (W. A. Kirklin, chairman).—This section has completed Proposed Tentative Methods of Testing Softening Point by Shouldered Ring and Ball Apparatus, which represents a suggested consolidation of the present Standard Method of Test for Softening Point of Bituminous Materials (Ring and Ball Method) (D 36 - 26) and the Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 - 42 T). This consolidated method is now being presented to the interested standing committees through their representatives on the section.

Section on Methods and Apparatus for Absolute Viscosity Measurements (W. F. Fair, Jr., chairman).—Descriptions of a number of methods describing apparatus and procedures for determining absolute viscosity have been obtained. These will be reviewed by the section and it is planned eventually to publish this information as a basis for the establishment of methods for absolute viscosity measurement.

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

Section on Pigment-Type Materials (C. E. Barnett, chairman).—Through its Task Group on Optical Microscope (R. P. Loveland, chairman), this section has completed a revision of the Tentative Method of Test for Particle Size Distribution of Subsieve Size Particulate Substances (E 20 - 33 T). This revision, in the form of procedures for the analysis by microscopic methods for the particle size distribution of particulate substances of subsieve sizes, has been approved by letter ballot of the section and of Technical Committee III. It is planned to present it to the Society through the standards Committee[2] for publication in the 1948 Supplement to the Book of Standards. While this revision of Method E-20 is too time-consuming for specification use, it can be used for calibration purposes, such as those involving turbidity, and is particularly useful for routine particle size measurement, especially in those cases where the material is of such shape that it does not approximately obey Stoke's law. It is suitable for a range of from 0.2 to 20 microns.

---

[2] See Editorial Note, p. 502.

Section on Sieves (A. A. Klein, chairman).—This section reviewed proposed tentative methods of test for sieve analysis of plastic calcined magnesia and of magnesium oxychloride composition, aggregates and fillers, prepared by Committee C-2. Pertinent comments and criticisms were made.

Technical Committee IV on Methods for Density (Wilmer Souder, chairman):

This technical committee has reviewed the following three methods for testing oxychloride cement compositions for bulk density, and a field method and a laboratory test for specific gravity of standard reference solutions. This review elicited a number of very pertinent comments and suggestions regarding these three methods. The technical committee has questioned the need for three separate methods and, among other things, has suggested that consideration be given to combining them.

Attention is being given to the need for a method to be used in standardizing powders used in insulation. This is particularly important where the insulation will be subject to vibrations of various frequencies, jars, etc. There was unanimous agreement that the use of small glass graduates such as had been suggested in previous recommendations and the employment of manual lifting and dropping operations were not satisfactory for use with those powders which are to be packed into relatively large spaces. The frictional retardation of the relatively large amount of wall surface on the glass graduate introduces and emphasizes effects which are no so prominent in practical conditions.

Since there has been no sustained interest in the development of a method for obtaining the bulk density of pharmaceutical powders, this subject is being dropped.

Technical Committee VII on Chemical Composition (G. E. F. Lundell, chairman):

This technical committee is reviewing two methods prepared by Committee C-2, namely, for analysis of magnesium chloride, and for sampling oxychloride compositions and ingredients. It also has under review some nine methods submitted by Committee D-19 on Industrial Waters.

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

This technical committee has recommended to Committee E-1 a revision in the Tentative Definitions with Procedures Relating to Conditioning and Weathering (E 41 – 42 T) which provides for changing the standard laboratory atmosphere to that having a relative humidity of 50 ± 2 per cent at a temperature of 23 ± 1.1 C. (73.4 ± 2 F.). This recommendation has been approved by letter ballot vote of the technical committee and after approval by the main committee will be presented to the Society through the Standards Committee[2] for publication in the 1948 Supplement to Book of Standards.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

Section A on Thermometers (R. M. Wilhelm, chairman) has continued to be very active and held meetings in Philadelphia, Pa., on September 18, 1947, and April 13, 1948. It has completed several specifications for new thermometers. These include three thermometers for measuring temperatures of bulk shipments of petroleum products, requested by Committee D-2 on Petroleum Products and Lubricants, also specifications for an A.S.T.M. bomb calorimeter thermometer, and an A.S.

T.M. Tag closed tester, low range thermometer. These will be presented to the Standards Committee[2] for inclusion in Tentative Specifications E 1.

A very thorough review of all of the thermometer specifications, which now total 55, has been made during the year. As a result, it is planned to recommend a number of minor changes in various thermometers to remove slight inconsistencies and in particular to further standardize all of the thermometers. Information on these proposed changes will appear in the A.S.T.M. BULLETIN.[3]

A third draft of Proposed Methods of Testing and Standardization of Liquid-in-Glass Thermometers is being studied.

Technical Committee XIII on Hydrogen Ion Determinations (E. B. Ashcraft, chairman):

This technical committee met in Washington during the week of March 1, 1948, at which time the Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T) was reviewed. Consideration was also given to a future program of work. These matters will be given further attention at a meeting to be held in June.

This report has been submitted to letter ballot of the committee, which consists of 12 members; 12 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

P. J. SMITH,
Secretary.

---

[3] "Changes Contemplated in Thermometer Specifications in 1949," ASTM BULLETIN, No. 155, December, 1948, p. 24.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative:

Specifications for A.S.T.M. Thermometers (E 1 - 47 T),
Method of Test for Particle Size Distribution of Subsieve Size Particulate Substances (E 20 - 33 T) in the form of a Recommended Practice for Microscopical Methods for the Particle Size Distribution of Particulate Substances of Subsieve Sizes (E 20 - 48 T), and
Definitions with Procedures Relating to Conditioning and Weathering (E 41 - 42 T).

These recommendations were accepted by the Standards Committee on September 29, 1948, and the revised tentatives appear in the 1948 Supplement to Book of A.S.T.M. Standards, Parts II, III-A, and III-B.

REPORT OF COMMITTEE E-2*
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held one meeting during the year at Atlantic City, N. J., on June 16 and 17, 1947. Meetings of all subcommittees and of the Advisory Committee were held at this time. A meeting of the Advisory Committee was held on November 14, 1947, at Pittsburgh, Pa.

During the past year, 9 new members were added to the committee and 2 members resigned, making a total membership of 102.

Committee activities were carried out through the subcommittees, all of which were active during the year. The work of the committee followed closely an activity schedule adopted by the Advisory Committee at its November meeting. The schedule set up projects under the direction of group leaders within each subcommittee to undertake the development of important phases of standardization in the field of spectrographic analysis.

The various subcommittees have been actively engaged in the collection of suggested analytical methods. These methods will be distributed to the membership of the entire committee for review, after which it is planned to publish these suggested methods in the form of a compilation which promises to be an important contribution toward standardization in this field of analysis. A total of 38 suggested methods have been received and additional methods are being solicited.

The present officers of the committee have been re-elected for the ensuing term of two years.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (R. W. Callon, chairman) prepared the first draft of a report on Location, Installation, and Safety Precautions for the Spectrographic Laboratory and it is being circulated within the subcommittee. A cooperative study on the quality of photographic materials is being made with the Eastman Kodak Co.

Subcommittee II on Fundamental Methods (E. B. Ashcraft, chairman) has carried out preliminary work on the writing of recommended procedures for measuring and specifying precision and accuracy for spectrographic methods of analysis.

Subcommittee III, Editorial (C. L. Guettel, chairman) has prepared a standard form for methods and edited several suggested methods. Its group on nomenclature has been active and the second report on nomenclature is ready for distribution.

Subcommittee IV on Standard Samples and Pure Materials (C. H. Corliss, chairman) has prepared a supplement[1] to the 1947 edition of the "Report on Standard Samples for Spectrochemical Analysis." This supplement brings up to date the records regarding the current availability of standards. A survey on the availability of pure materials of importance to spectrographic analysis is under way. The development of standard shapes and sizes for graphite electrodes has been started.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] ASTM BULLETIN, No. 157, March, 1949.

This report has been submitted to letter ballot of the committee, which consists of 102 members; 65 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. F. SCRIBNER,
Chairman.

MARY E. WARGA,
Secretary.

REPORT OF COMMITTEE E-3*

ON

CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held two meetings during the year: at Atlantic City, N. J., on June 17, 1947, and at Washington, D. C., on March 5, 1948, during the spring group committee meetings. The work of the committee has centered around laying the foundations for issuing new and revised methods in time for a new edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals. This work is in the hands of subcommittees which in several cases are carefully restudying the procedures which have already been published in order to keep them up-to-date. New methods are being submitted to letter ballot as fast as they become available. Twenty-three divisional and subcommittee meetings were held at Atlantic City and at Washington. In addition special committees and informal groups have met to discuss separate phases of committee work.

Important progress has been made in the work of Division D on General Analytical Methods. Four subcommittees are now organized and functioning. This division each year has sponsored a symposium on some special technique. The symposium at Washington was concerned with the use of organic reagents in analysis.

The Advisory Committee held two important meetings: on June 17, 1947, in Atlantic City and on March 4, 1948, in Washington. At these meetings the broad policies to be followed in the program of Committee E-3 were carefully discussed and action was taken to expedite the work of the committee.

The Advisory Committee recommended that the Tentative Method for Sampling Molybdenum Salts and Compounds for Metallurgical Use (E 66 – 34 T) be transferred to another committee because it does not fall within the scope of Committee E-3. Following this recommendation, Committee A-9 on Ferro-Alloys has assumed responsibility for this method.

The Advisory Committee in considering the functions of Division D has established the standard practice of having all new procedures reviewed by this division in order that specifications for the necessary apparatus and reagents can be drawn up. Methods are to be submitted to Division D when they are in final substance and this may be before final editing has been completed.

The By-laws of Committee E-3 have been amended, and approved by letter ballot, in order to provide for the submission of annual reports by division and subcommittee chairmen at a sufficiently early date to permit the secretary of Committee E-3 to prepare the report of the committee in advance of the annual meeting. An amendment was also approved providing for the representative of Committee E-2 on Spectrographic Analysis, who is not necessarily its chairman, to be a member of the E-3 Advisory Committee.

Seventeen new members were added to the committee during the year, five members resigned, and one member died. The active membership of the committee is now 97.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Honorary Chairman, G. E. F. Lundell.
Chairman, D. R. Evans.
Vice-Chairman, C. B. Francis.
Vice-Chairman, H. A. Bright.
Secretary, J. W. Stillman.
Members-at-large of Advisory Committee:
H. Kirtchik, R. P. Nevers.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1947 Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the proposed Tentative Photometric Method for the Determination of Boron in Steel. The new tentative method was accepted by the Standards Committee on March 5, 1948, and has been assigned the designation E 30 – 48 T.

ADOPTION OF TENTATIVES AS STANDARD

Committee E-3 recommends that the following five tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of:

Chemical Analysis of Metallic Materials for Electrical Heating (E 38 – 42 T),
Chemical Analysis of Copper (Electrolytic Determination of Copper) (E 53 – 46 T),
Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (E 55 – 46 T), with the following editorial revisions:

Section 4 (*a*).—Delete the words "with shears" at the end of the first sentence. In the second sentence, change "0.010 in." to read "0.040 in."

Section 6 (*a*).—Change to read: "The prepared sample shall weigh three times the amount required for the analysis and shall be divided into three equal parts, each of which shall be placed in a package and sealed, one for the manufacturer, one for the purchaser and one for an umpire, if necessary."

Sampling Slab Zinc (Spelter) (E 65 – 46 T), and

Tentative Recommended Practices for:

Apparatus and Reagents for Chemical Analysis of Metals (E 50 – 46 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the remaining tentatives which have stood for two years or more without revision be retained in their tentative status for further study.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division A on Ferrous Metals (Arba Thomas, chairman).—The Ferrous Division held only one meeting during the year at the Annual Meeting of the Society at Atlantic City in June, 1947. Another meeting was scheduled for the 1948 spring group committee meetings in Washington, D. C., but was cancelled due to the illness of the chairman. The June, 1947, meeting was well attended and considerable interest was shown by all present.

The subgroup under C. Ruhe and E. J. Dunn on the determination of aluminum in alloy steel has circulated samples of steels of the stainless type for analysis by members of this subgroup. This analytical work has not yet been completed.

---

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

The subgroup responsible for the development of a satisfactory method for the determination of beryllium in alloys of the turballoy type has not made any further progress during this year. A method, however, has been proposed which from all standpoints appears to be quite satisfactory.

During the year considerable work was done on a critical survey of the present published methods with the object of bringing them up-to-date and taking advantage of any new analytical techniques that are available. It is hoped that this work can progress more quickly in the near future.

The Division recommended that Tentative Methods E 38 - 42 T be adopted as standard.

Divison B on Non-Ferrous Metals (D. R. Evans, chairman).—This division and its subcommittees held two meetings during the year, one at Atlantic City, in June, 1947, and the other at Washington, in March, 1948. Two local informal sessions were also held. A meeting of the Joint Subcommittee on Analysis of Powdered Metals, of Committee B-9 on Metal Powders and Metal Powder Products and Committee E-3, was held in October, 1947, and plans outlined for the work to be undertaken.

Subcommittee B-1 on Copper and Its Alloys (R. P. Nevers, chairman) is continuing its work on the preparation and testing of methods for nickel silver.

Subcommittee B-2 on Lead, Tin, Antimony, Bismuth and Their Alloys (C. I. Luke, chairman) is investigating the possibilities of improving the present procedures for the analysis of pig lead.

Subcommittee B-3 on Aluminum and Magnesium and Their Alloys (H. V. Churchill, chairman) has prepared and furnished two samples of aluminum for cooperative work with the photometric subcommittee.

Subcommittee B-6 on Nickel and Its Alloys (C. Sterling, chairman) is cooperating with an electronic nickel subcommittee on the preparation and testing of methods for the analysis of nickel used in vacuum tubes. Methods for the analysis of high nickel-copper alloys are being considered and cooperative work on a Bureau of Standards sample is planned.

Subcommittee B-7 on Photometric Methods (C. Zischkau, chairman) has completed its work on a photometric method for cobalt which will be submitted to the Society for publication as tentative. Cooperative work has been done on photometric procedures for silicon, iron, copper, nickel, chromium, manganese, titanium, bismuth, and magnesium in aluminum and aluminum alloys. Procedures are being prepared and it is hoped that these can be completed for the next edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals.

Subcommittee B-8 on Polarographic Methods (W. W. Braun, chairman) has done some cooperative work on the polarographic analysis for cadmium and lead in a sample of zinc furnished by R. W. Coltman. Mr. Coltman has also prepared an extensive Bibliography on Polarography which has been submitted to A.S.T.M. Headquarters for consideration for publication.

Division C on Sampling (W. C. Bowden, Jr., chairman).—The Sampling Division held one meeting during the year at Atlantic City, in June, 1947. Eleven sampling procedures have been revised and approved by the Division.

New specifications for the sampling of pig lead have been written in cooperation with Subcommittee II of Committee B-2 on Non-Ferrous Metals and Alloys.

The Division has recommended that Tentative Methods E 55 - 46 T and E 65 - 46 T be adopted as standard.

Division C has been represented on the

Joint Subcommittee on Analysis of Powdered Metals, of Committees B-9 and E-3, and expects to confer further on sampling procedures as applied to powdered metals.

Committee B-5 on Copper and Copper Alloys has asked for a discussion of cooperative work on sampling. Division C plans to meet with this committee and offer its services on revising present sampling procedures.

Division D on General Analytical Methods (S. E. Q. Ashley, chairman).—The General Analytical Division has held two meetings during the year, one at Atlantic City, in June, 1947, and one at Washington, in March, 1948. Division D also sponsored a discussion of "Precision and Accuracy" at Atlantic City and a successful Symposium on Organic Reagents in Metal Analysis at Washington at which the following papers[2] were presented:

"Organic Reagents for Gravimetric Analysis," by John F. Flagg,

"The Use of Organic Solvents in Metal Analysis," by C. J. Rodden,

"Organic Reagents in Colorimetric Analysis," by John H. Yoe,

"Organic Reagents for Volumetric Analysis," by John F. Flagg, and

"Oxidation-Reduction Indicators," by G. Frederick Smith.

The Division is considering sponsoring other symposiums in the future.

The organization of four subcommittees has been completed and these subcommittees are now actively at work.

Subcommittee D-1 on Reagents (T. C. Bryson, chairman) has been engaged in the following projects:

(*1*) Preparation of a list of all reagents and chemicals used in the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals.

(*2*) Expansion of Section 6 on hazardous chemicals of the Tentative Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (E 50 – 46 T) to include perchloric acid, ether, hydrogen sulfide, and various other reagents,

(*3*) Inclusion in E 50 of a table showing color charts and pH range of indicators,

(*4*) Establishment of size distribution limits for calcium chloride used as a drying reagent,

(*5*) Development of a method of analysis and limits for boron in phosphoric acid,

(*6*) Establishment of limits for lead in liquid bromine,

(*7*) Preparation of statements explaining the dilution of stronger normal solutions to obtain weaker solutions, and

(*8*) A program of checking various chemicals to see if they meet the reagent standards of the American Chemical Society.

Subcommittee D-2 on Apparatus (A. H. Bushey, chairman) has been engaged in the following projects:

(*1*) Preparation of revisions of the sections on potentiometric apparatus and the mercury cathode and the development of a new section describing apparatus for the dead-stop end-point determination of Tentative Recommended Practices E 50,[3]

(*2*) Consideration of performance specifications of analytical balances,

(*3*) A study of requirements to be included in specifications for analytical filter papers, this project to be carried out in cooperation with Committee D-6 on Paper and Paper Products, and

(*4*) Review of the requirements for the apparatus for determination of boron in steel.

Subcommittee D-3 on Procedures (J. H. Harley, chairman) was formed in June, 1947, to advise Committee E-3 on matters of common procedures used in the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals. The aim is to reconcile differences in the descriptions and the actual details of these common procedures. An outline of the procedures used has been prepared and distributed to the membership. Individual members are working on sections of the outline, determining what the differences are. Recommendations will be made at the completion of the work.

Subcommittee D-4 on Statistical

---

[2] Abstracts of these papers were published in the ASTM BULLETIN, No. 151, March, 1948, p. 31.

[3] See Editorial Note, p. 509.

Methods (G. Wernimont, chairman) held a meeting in June, 1947. The procedure for conducting cooperative tests has been held up until Committee E-11 on Quality Control of Materials finishes work on a Recommended Practice for Interlaboratory Test Programs. The chairman of Subcommittee D-4 is serving on the Task Group of Committee E-11.

This report has been submitted to letter ballot of the committee, which consists of 97 members; 84 members returned their ballots, of whom 79 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

J. W. STILLMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the recommendation that the Recommended Practices for Apparatus for Chemical Analysis of Metals be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 29, 1948, and the new tentative recommended practice appears as a separate reprint bearing the designation E 50 - 48 T.

REPORT OF COMMITTEE E-4*
ON
METALLOGRAPHY

Committee E-4 on Metallography and its subcommittees held meetings during spring group committee meeting week in Washington, D. C., on March 1 and 2, 1948.

Two newly formed subcommittees, II on Definitions and V on Microhardness, held their initial meetings in Washington and have inaugurated their respective programs.

Standardization of the use of the electron microscope including sample preparation will be sponsored by Committee E-4. Subcommittee I on Selection and Preparation of Samples and Subcommittee IV on Photography will act jointly on this standardization project after their membership is expanded to include specialists in the fields of sample preparation and instrument use.

On recommendation of the Nominating Committee (W. L. Fink, chairman) the present officers of Committee E-4 were reelected to their present positions.

The membership of Committee E-4 has increased during the past year to a total of 86 members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following four tentatives be continued in their present status without revision:

Tentative Methods of:

Preparation of Micrographs of Metals and Alloys (E 2 - 44 T),

Preparation of Metallographic Specimens (E 3 - 46 T), and

Tentative Recommended Practices for:

Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 - 46 T), and

Determining the Inclusion Content of Steel (E 45 - 46 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (J. R. Vilella, chairman) reviewed Tentative Methods E 3 and recommended that they be continued as tentative without change.

Subcommittee II on Definitions (P. A. Beck, chairman) held its first meeting, mapped out its initial program and has made task assignments for the preparation of first drafts of definitions in the various phases of metallography with which Committee E-4 is concerned.

Subcommittee IV on Photography (L. V. Foster, chairman) reviewed Tentative Methods E 2 - 44 T and recommended that they be continued as tentative without change.

This subcommittee arranged the Symposium on Color Metallography[2] being presented at this Annual Meeting of the Society. In addition, this group has sponsored and arranged for the photomicrographic exhibits which form such an interesting portion of the current

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[2] To be issued as a separate publication.

Photographic Exhibit at the Annual Meeting of the Society.

Subcommittee V on Microhardness (Alexander Gobus, chairman) had its first meeting in Washington at which time the various phases of microhardness testing as they pertain to metallography were discussed. Consideration was given to the types of loading and indenters associated with existing instruments, the accuracy of measuring diamond impressions, the effects of grain orientation on these impressions, etc. Tasks were assigned for preliminary surveys to obtain data relative to several of the foregoing items. Results are to be reported at the second meeting of the subcommittee to be held during the Annual Meeting.

Subcommittee VI on X-Ray Methods (W. L. Fink, chairman) reviewed the Tentative Recommended Practice E 43 – 46 T and recommended its continuation as tentative without change.

This group is also completing the work on recommended practices for back-reflection determination of crystal orientation and for the determination of preferred orientation in worked materials.

Subcommittee VI on X-Ray Methods has been assigned the work of requisite standardization in microradiography as a metallographic tool. Since Committee E-7 on Radiography has jurisdiction over microradiography as a radiographic tool, adequate liaison is to be established with Committee E-7.

Joint committee work in collaboration with the American Society for X-Ray and Electron Diffraction is resulting in new and much more informative index cards applicable to Tentative Recommended Practice E 43.

Subcommittee VIII on Grain Size (M. A. Grossman, chairman).—Section B on Ferrite Grain Size (R. E. Penrod, chairman) has made considerable progress toward the establishment of a recommended practice for the determination of ferritic grain size and expects to have the first draft of this proposal prepared in the immediate future.

Section C on Non-Ferrous Grain Size (C. H. Samans, chairman) has been correlating the grain size measuring practices currently applied to several non-ferrous metals. The chairman is optimistically approaching a general solution which will apply in all cases in the non-ferrous field and will integrate with the ferrous grain size measuring procedures. The specific applications at present concern aluminium magnesium, nickel, and brass.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) reviewed the Tentative Recommended Practice E 45 and unanimously recommended the continuation of this tentative without change.

This report has been submitted to letter ballot of the committee, which consists of 86 members; 67 members returned their ballots, of whom 66 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY NORTON,
Secretary.

REPORT OF COMMITTEE E-5*
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION

Committee E-5 on Fire Tests of Materials and Construction held one meeting during the past year in Washington, D. C., on March 5, 1948. Meetings of the various subcommittees preceded this meeting.

The committee now consists of 53 members of whom 22 are classified as producers, 9 as consumers, 16 as general interest members, and 6 as consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. L. Brown.
Vice-Chairman, Wm. J. Krefeld.
Secretary, Hugh M. Robinson.

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 47**).[1]—The committee recommends the following tentative revision of Standard Methods E 119, which proposed changes reflect the general practice of the laboratories concerned with fire tests as regards the number of thermocouples used and the frequency of readings. From the nature of the changes it can be assumed that they will not invalidate the results of tests made according to the present requirements, even if the minimum number of thermocouples and the maximum spacing of readings therein given were used.

Section 3(*a*).—In the first sentence change "five thermocouples" to read "nine thermocouples."

Section 3(*b*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 3 (*b*). The temperatures shall be read at intervals not exceeding 5 min. during the first [hour] *two hours*, and thereafter the intervals may be increased to not more than [15] *10* min.

The other proposed revision waives the requirement for applying a correction to the resistance period for variation of intensity of furnace exposure from that prescribed where the correction will not change the fire resistance classification of the construction tested, as expressed in time periods taken at intervals of a given number of minutes, fractions of an hour, or hour, apart. For the purpose the following tentative revision is proposed:

Section 5(*b*).—Change the first part of the first sentence to read as follows, by the addition of the italicized words and the omission of the word in brackets:

> When the indicated resistance period is ½ hr. or over, [and] determined by the average or maximum temperature rise on the unexposed surface or within the test sample, or by failure under load, a correction shall be applied for variation of the furnace exposure from that prescribed, *where it will affect the classification*, by multiplying the indicated period....

Standard Method of Test for Combustible Properties of Treated Wood by the Crib Test Method (**E 160 - 46**).[2]—As indicated in the 1946 Report[3] when this test method was recommended for adoption as standard, and in Section 3 thereof, the sampling procedure applies for the condition of penetration of treating solution throughout the section and is generally applicable for trim and other stock in the smaller range in thickness. In the 1947 Report[4] the need was indicated for a sampling procedure for wood which has been given impregnation treatment with fire-retarding chemicals that in significant amounts do not penetrate the full section. This would apply, in general, for stock of 2-in. or greater thickness used in building construction or scaffolding without further ripping or planing.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[2] 1946 Book of A.S.T.M. Standards, Part II.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 293 (1946).

The work in the committee has now progressed to the point where such a sampling procedure can be submitted for the condition where arrangements can be made so the sampling can be done at the treating plant from each charge being treated. Accordingly, the following tentative revision of Standard Method E 160 is proposed:

New Section.—Add a new Section 4 to read as follows, renumbering the subsequent sections accordingly:

4. ***Sampling for Surface-Impregnated Wood.***—Where arrangements are made for sampling of each charge at the treating plant, the specimens shall be obtained as follows:

(*a*) One sample piece furnished by the purchaser, previously agreed upon, selected and identified, shall be included for each **5000** bd. ft. in a charge, but no charge shall contain less than 4 nor more than 8 sample pieces. These sample pieces shall be of the same species of wood as the rest of the material in the charge and shall be fairly representative of the lot with respect to rate of growth, density, heartwood, and sapwood. They shall be identified by metal tags or the equivalent.

(*b*) Placement of sample pieces in the material to be treated on a tram shall be representative of the average exposure to the treating solution and yet be accessible for ready removal without seriously disturbing the loading.

(*c*) Sample pieces shall be at least 6 ft. in length and may be either full-size rough or dressed standard. They shall be of the same cross-sectional dimensions as the material in the lot for thicknesses up to and including 3-in. in nominal thickness. Where material in the charge is over 3 in. in thickness, irrespective of width, sample pieces shall be 4 by 4 in. in nominal size.

(*d*) One set of crib test specimens shall be cut from each selected sample piece and shall consist of 24 pieces ½ by ½ in. in cross-section and 3 in. in length, smooth-sawed to dimensions within plus or minus $\frac{1}{32}$ in. Not less than four similar pieces shall be cut for moisture test specimens. The specimens shall not be cut closer than 2 ft. from the ends of sample pieces nor closer than ½ in. from their edges. The specimens shall be cut so that one side will be from a treated face of the sample piece and shall be cut or chosen from locations fairly representative of density, heartwood, and sapwood for the piece as a whole.

Standard Method of Test for Combustible Properties of Treated Wood by the Fire-Tube Test Method (**E 69 – 47**).—Another sampling procedure for this method was also developed for surface-treated timber or lumber similar to that given above for the tentative revision of Standard Method E 160. The committee therefor recommends the following tentative revision for Standard Method E 69:

New Section.—Add a new Section 4 to read the same as that recommended above for Section 4 of Method E 160 except that in Paragraph (*c*) the length of the sample pieces shall be 8 ft. instead of 6 ft. and Paragraph (*d*) shall read as follows:

(*d*) Two fire-tube test specimens shall be cut from each selected sample and shall be ¾ by ¾ in. in cross-section and 42 in. in length, smooth-sawed to dimensions within plus or minus $\frac{1}{32}$ in. A 2-in. length thereof shall be cut off for moisture test specimens. The specimens shall not be cut closer than 2 ft. from ends of sample pieces nor closer than ½ in. from their edges. The specimens shall be cut so that one side ¾ in. in width will be from a treated face of the sample piece and shall be cut or chosen from locations fairly representative of density, heartwood, and sapwood for the piece as a whole.

The recommendations in this report

---

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 436 (1947).

have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

GENERAL ACTIVITIES OF THE COMMITTEE

Fire Tests of Acoustical and Similar Finishes.—Additional data from fire hazard tests by several methods have become available during the year, particularly as concerns effect of fire-retarding surface finishes. Considerable work has been done on classifications of such materials from the fire hazard standpoint and applications thereof to building occupancies in connection with the development of city and recommended general building code requirements. These all emphasize the need for a standardized test method or methods on which the criteria for performance for the different classifications can be based.

Nomenclature and Definitions.—The work for the year has been concerned mainly with the development of a definition and method of test for incombustibility of materials. Improvements have been made in the British apparatus for incombustibility tests according to British Standard BS476-1932, and results of exploratory tests with this apparatus have been reported before the Society.[6] The improvements comprise the addition of an inner tube to promote temperature uniformity axially and radially, and the omission of the observation chamber below the furnace tube, since little or no use has been found for this detail. Some equipments have been constructed with the revised plan and several more are due to be made and applied in tests with a typical range of materials.

Fire Tests of Roof Coverings.—In conformity with the general plan of other testing procedures standardized by the committee, an effort is being made to define and standardize the tests required for determining the pertinent properties of roofing materials so that they will indicate more or less continuous scales of performance. These could then be applied in connection with classifications to be defined by those adopting the method as a basis for performance standards. This would obviate any need for the committee to develop classifications and define the performance required for the different classes.

Review of Standards and Definitions.—To facilitate the review of standards, test methods, and definitions relating to fire resistance and fire hazard, developed by other A.S.T.M. committees and referred to this committee for comments, as well as for consideration of the general technical aspects of other matters requiring attention, an advisory committee has been formed consisting of the officers of the committee and chairmen of the subcommittees.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 29 members returned their ballots, of whom 24 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

S. H. INGBERG,
Chairman.

H. M. ROBINSON,
Secretary.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[6] *Proceedings*, Vol. 45, p. 866 (1945).

REPORT OF COMMITTEE E-6*

ON

METHODS OF TESTING BUILDING CONSTRUCTIONS

Committee E-6 on Methods of Testing Building Constructions and six of its eight subcommittees held meetings on June 18 and 19, 1947, at Atlantic City, N. J., during the Annual Meeting of the Society. Progress was reported in various aspects of the committee's activities. Further meetings of the committee and five of its subcommittees were held in Washington, D. C., on March 2 and 3, 1948, during Spring Committee Week.

At the present time the committee consists of 44 members and 2 consulting members. The members are not classified.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. A. Liska.
First Vice-Chairman, F. E. Richart.
Second Vice-Chairman, K. F. Wendt.
Secretary, J. H. Courtney.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Panels for Light-Building Construction (H. L. Whittemore, chairman) developed the Tentative Methods of Conducting Strength Tests of Panels for Building Construction (E 72 – 47 T). These methods will be considered by the committee for further action at its next meeting.

Subcommittee II on Connections and Assembled Structures (G. M. Rapp, chairman) has three projects under consideration. These involve methods of testing joints between prefabricated panels to determine the strength of the connections laterally and in shear.

Subcommittee III on Large Structural Units (F. E. Richart, chairman) during the past year has been engaged in the preparation of methods of testing truss assemblies. It is expected that these methods will be submitted to the Society for publication as tentative during 1948.[1] Tests of large structural units are not commonly required as acceptance tests but are occasionally required for built-up structures in which the behavior of composite members, the effect of special connections, and details of fabrication cannot be predetermined by theoretical analysis. For that reason, it is the object of this subcommittee to develop standardized testing methods and techniques for large structural units.

Subcommittee IV on General Structural Elements (K. F. Wendt, chairman) reports progress on the development of methods of testing prefabricated floor and roof constructions. A preliminary draft of a proposed tentative has been circulated to the subcommittee for comment.

Subcommittee V on Durability (Weathering, Exposure and Moisture Penetration) (D. E. Parsons, chairman) reviewed many of the phases of the general problem of durability of buildings and the accomplishments of other groups relating to them. It was considered that other A.S.T.M. committees are responsible for methods of testing individual materials and that other subcommittees are better qualified to deal with problems of ventilation, moisture condensation, and vapor barriers. The development of methods of testing the resistance to rain penetration of panels representing exterior wall and roof construction is being undertaken, and studies are being made of the feasibility of preparing methods for rating the durability of composite elements of buildings under limited conditions of exposure.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] See Editorial Note, p. 516.

Subcommittee VI on Insulation (F. G. Hechler, chairman) held its organization meeting in June, 1947. It was agreed that a primary need was the development of a simple and inexpensive method for checking the thermal performance of structures in the field. The subcommittee efforts have been directed toward obtaining information on such methods.

Subcommittee VII on Sound Transmission (R. K. Cook, chairman) held its organization meeting in June, 1947. Since then a draft of a Tentative Test Code for the Laboratory Measurement of Airborne-Sound Transmission Loss of Building Floors and Walls has been prepared and considered by the subcommittee in meeting. This draft will be circulated for further review and comment.

Subcommittee VIII on Fire Resistance (Harold Perrine, chairman) has been largely engaged in a review of the work of Committee E-5 on Fire Tests of Materials and Construction to determine whether the program of that committee is adequate to meet the needs of Committee E-6. One point of interest to the committee is the application of the proposed work of Committee E-5 to small constructions, such as prefabricated panels. The practicability of establishing a less severe time-temperature curve than the present standard for testing prefabricated panels, and applicable to small buildings, is to be studied by the subcommittee.

This report has been submitted to letter ballot of the committee which consists of 44 members; 41 members returned their ballots, of whom 38 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

J. H. COURTNEY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-6 presented to the Society through the Administrative Committee on Standards the recommendation that the Methods of Testing Truss Assemblies be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 29, 1948, and the new tentative methods appear in the 1948 Supplement to Book of A.S.T.M. Standards, Part II, bearing the designation E 73 - 48 T.

REPORT OF COMMITTEE E-7* ON RADIOGRAPHIC TESTING

Committee E-7 on Radiographic Testing met at Detroit, Mich., on June 21, 1948, in conjunction with the Annual Meeting of the Society.

The major effort of the committee during the preceding year was devoted to the project of expanding the scope of the committee to include other non-destructive tests in addition to radiography. A letter ballot dated July 16, 1947, showed the membership to be overwhelmingly in favor of such a step. On September 25, 1947, preliminary reaction of the Board of Directors was reported to be sympathetic but concerned lest the new scope infringe on the jurisdiction of other committees.

The Executive Council of the committee considered this problem in meetings held on October 22, 1947, March 1, 1948, and June 21, 1948. A special subcommittee on the scope and by-laws of the committee, under the chairmanship of J. H. Bly, was appointed at the first of these meetings. This subcommittee prepared a complete new set of by-laws, changing the name of the committee to "Committee E-7 on Non-Destructive Testing," and revising the scope clause. These were presented to the Council at the March meeting and further revised. They were then approved by the committee by letter ballot dated March 25, 1948. At the June meeting of the committee, it was decided to submit the revised scope clause, together with an acceptable alternate wording, to the Board of Directors for approval.

In conjunction with the June, 1948, meeting, the committee sponsored an Informal Round Table Discussion on Ultrasonic Testing, as a preliminary venture beyond the radiographic field. This discussion assumed unexpected proportions when an attendance of approximately 200 was realized. Historical and theoretical reviews of the method, presented by C. H. Hastings, of Watertown Arsenal, and Louis Gold, of Brown University, respectively, were followed by a lively discussion of practical applications by more than a score of participants. The stenographic transcript of this discussion is being considered for publication or reproduction, and it is hoped to publish Messrs. Hastings' and Gold's presentations. In view of the outstanding success of this discussion the committee hopes to present a more formal symposium on ultrasonic testing in conjunction with next year's meeting.

On January 25, 1948, the Administrative Committee on Standards accepted the recommendation of the committee that the Tentative Methods of Radiographic Testing of Metal Castings (E 15 – 39 T) be withdrawn as obsolete.

Preparation of revisions of the Tentative Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (E 52 – 45 T) is still in process.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

The committee was advised by H. H. Lester, who had been chairman of Committee E-7 since its organization in 1938, that he did not wish to be considered as a candidate for re-election. The following officers were accordingly nominated and elected for the ensuing term of two years:

Chairman, J. H. Bly.
Vice-Chairman, H. C. Amtsberg.
Secretary, D. T. O'Connor.

The committee desires to record its sincere thanks to Mr. Lester for his long and distinguished service to the committee as its chairman since its organization.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

D. T. O'CONNOR,
Secretary.

REPORT OF COMMITTEE E-8*
ON
NOMENCLATURE AND DEFINITIONS

Committee E-8 on Nomenclature and Definitions was relatively inactive during the war years. This lessening of activity is understandable due largely to the fact that there were much more urgent problems concerning specifications and methods of test for engineering materials. Accordingly, this is the first report to the Society since 1944.

A meeting of the Advisory Committee was held in New York City on April 20, 1948, at which time the work of the committee was reviewed and plans made for completing or reactivating various matters before the committee and its several subcommittees.

In 1946 the following appointments to the Advisory Committee, each for terms of three years, were made by the Board of Directors:

J. W. McBurney, and G. B. Waterhouse, from 1946 to 1949.

J. L. Miner, and C. A. Marlies, from 1947 to 1950.

P. V. Faragher and L. H. Fry, from 1948 to 1951.

In 1947, P. J. Smith was designated by the Executive Secretary of the Society to serve as secretary ex-officio of the committee in place of Mr. R. E. Hess who had served the committee in this capacity from 1926.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, P. V. Faragher.

Vice-Chairman, C. A. Marlies.

Committee E-8 suffered a severe loss in the sudden death of Cloyd M. Chapman on July 2, 1944. Apart from his interest in the administrative and technical affairs of the Society, he was deeply interested in the niceties of English expression which fitted him admirably for the chairmanship of Committee E-8, in which capacity he served from the organization of the committee in 1920. His loss has been greatly felt by the committee and all those who were fortunate enough to know him.

NEW TENTATIVE DEFINITIONS

There is a continuing interest on the part of technical committees in the establishment of definitions of terms applying to materials in their particular field of activity. This is particularly true in the case of new committees, a number of which have been organized by the Society in the past several years. At the 1948 Annual Meeting, the following definitions were submitted for publication as tentative:

Tentative Definitions of:

Terms Relating to Natural Building Stone, prepared by Committee C-18 on Natural Building Stones,

Terms Relating to Paint and Paint Materials (D 16 - 48 T), comprising seven new definitions prepared by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Terms Relating to Textile Materials (D 123 - 48 T), comprising five revised definitions and 13 new terms prepared by Committee D-13 on Textile Materials, and

Terms Relating to Thermal Insulating Materials (C 168 - 47) which includes five revised definitions proposed by Committee C-16 on Thermal Insulating Materials.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

Committee E-8 is planning during the coming year to review each of these definitions and submit to the sponsoring committee any recommendations for their improvement which may result from this review.

In addition to the above, there are individual definitions appearing in several new and revised methods being submitted as tentative this year. These are not listed in detail, but will also be reviewed in Committee E-8, together with the definitions listed above.

Committee E-8 has also received for review Proposed Tentative Definitions of Terms Relating to Shipping Containers prepared by Committee D-10 on Shipping Containers. Also, a set of definitions relating to adhesives has been completed by Committee D-14 on Adhesives as an addition to the Tentative Definitions of Terms Relating to Adhesives (D 907 – 47 T). Both of these series of definitions will be reviewed in Committee E-8.

Committee E-8 has under review an extensive list of Definitions of Terms Relating to Powder Metallurgy prepared by Committee B-9 on Metal Powders and Metal Powder Products. A number ofcomments on these definitions have been received and these will be discussed with Committee B-9 prior to publication of the definitions as tentative.

NEW AND REVISED STANDARD DEFINITIONS

The following recommendations for adoption of definitions as standard were presented at the 1948 Annual Meeting:

Tentative Definitions of Terms Relating to:

Concrete and Concrete Aggregates (C 125 – 46 T), proposed by Committee C-9 on Concrete and Concrete Aggregates,

Gypsum (C 11 – 41 T), proposed by Committee C-11 on Gypsum,

Petroleum (D 288 – 47), addition of new definition of "precipitation naphtha," proposed by Committee D-2 on Petrolum Products and Lubricants,

Thermal Insulating Materials (C 168 – 47), adoption of definition of "molded type pipe insulation," proposed by Committee C-16 on Thermal Insulating Materials,

Magnetic Testing (A 127 – 46), adoption of definitions: Aging-magnetic, demagnetizing coefficient, magnetic-instability and permeability space, proposed by Committee A-6 on Magnetic Properties,

Soaps and Other Detergents (D 459 – 47 T), proposed by Committee D-12 on Soaps and Other Detergents,

Textile Materials (D 123 – 47), adoption of definitions for azlon, duck, and woven narrow fabric, proposed by Committee D-13 on Textile Materials, and

Naval Stores and Related Products (D 804 – 46 T), proposed by Committee D-17 on Naval Stores.

The proposed revised definition of the term slate submitted by Committee C-18 on Natural Building Stones has been approved by Committee E-8. In voting on this revised definition a number of suggestions for minor changes have been received and these have been brought to the attention of the sponsoring committee.

The Definitions of Terms Relating to Gypsum contain a definition for the term aggregate, which term is of interest to a number of other committees. Committee E-8 has accordingly requested Committee C-11 on Gypsum to defer this particular recommendation for a year in order to provide opportunity for review and agreement on a definition for this term that would be acceptable to all committees concerned.

The other terms being recommended for adoption this year will be reviewed by Committee E-8 immediately after the Annual Meeting and any comments and suggestions will be presented to the originating committee.

ACTIVITIES OF SUBCOMMITTEES

The E-8 Subcommittees on Definitions of Sand and Aggregate, on Definition of

Sieve and Screen, and on Definition of Concrete have been without a chairman since the death of Mr. Chapman. The Advisory Committee is endeavoring to obtain chairmen for these three subcommittees and it is expected the work on these particular subjects will accordingly be renewed during the coming year.

The Subcommittee on Definitions Relating to Bituminous Materials (K. G. Mackenzie, chairman) reviewed a number of new and revised definitions for bituminous road materials prepared in Committee D-4 on Road and Paving Materials, and for bituminous waterproofing materials from Committee D-8 on Bituminous Waterproofing and Roofing Materials. The suggestions received were submitted to the originating committees. Committee D-4 has since revised certain of the definitions and these are now being reviewed. These new and revised definitions are to be issued as a tentative revision of the Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 - 46).

The Subcommittee on Definition of Terms Relating to Heat Treatment (L. H. Fry, chairman) has reviewed the revised definitions prepared by the Joint Committee, which contains representatives from the American Society of Metals, Society of Automotive Engineers, American Foundrymen's Society, and A.S.T.M. The Editorial Committee of the Joint Committee is now completing a review of certain definitions, following which it is expected that the revised definitions will be ready for consideration by Committee E-8 as a revision of the Standard Definitions of Terms Relating to Heat Treatment of Metals (E 44 - 43).

The work of the Subcommittee on Definitions of Net and Gross Calorific Values of Fuels has about been completed with the adoption as standard of the Definitions of The Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 - 44). The members of this subcommittee, however, will be canvassed to determine if there are any other projects that need attention. If not, this subcommittee will be discontinued.

The same is also true of the Subcommittee on the Definition of Glass which reviewed and approved the Standard Definition of the Term Glass (C 162 - 47) which was issued as tentative in 1941, revised in 1945 and adopted as standard in 1947.

The Advisory Committee decided to reactivate the subcommittee on Coke, and a further effort will be made to prepare a definition of coke that will be acceptable to the various interested committees.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

P. V. FARAGHER,
Chairman.

P. J. SMITH,
Ex-Officio Secretary.

REPORT OF COMMITTEE E-9[*]
ON
FATIGUE

During the past year, most of the activity of Committee E-9 on Fatigue has been directed toward preparation of the Manual on Fatigue Testing. Since the committee is frequently asked when the Manual will be available and has had quite a number of requests for information of the kind which it now has, the present status of this project in detail is outlined as follows:

1. Introduction	2nd revision.	Complete
2. Nomenclature	4th revision.	Complete
3. Machines	1st revision.	Material to be Added
4. Specimens and Preparation	3rd revision.	Complete
5. Test Procedures and Technique	1st revision.	Complete
6. Presentation of Data	3rd revision.	Complete
7. Interpretation of Data	1st revision.	Complete
8. Bibliography	1st revision.	Complete

The manuscripts now total 167 pages, including the figures. The new material was reviewed at the 1948 Annual Meeting in Detroit and it is hoped that a final manuscript will be completed before the end of the year. Since it is apparent that it will be some time before the published Manual is available, the committee will try to be helpful during the interim in answering specific questions coming within the scope of the project.

One of the Manual sections (Nomenclature) will be published in the A.S.T.M. Bulletin[1] in August in order to have the benefit of discussion from the A.S.T.M. membership at large. Comments should be sent to J. M. Lessells at Massachusetts Institute of Technology.

Another document, a tentative Approximate Statistical Method for Fatigue Data, has been under consideration relative to the Presentation of Data section, but due to its controversial nature has been submitted to Committee E-11 on Quality Control of Materials. After review by that committee and further revision by Committee E-9 it will be published in the Bulletin to obtain discussion from the general A.S.T.M. membership. Comments should be sent to the Chairman of E-9.

The activities of Committee E-9 other than the Manual have comprised the following:

1. Review of papers and sponsoring of sessions on fatigue.
2. Discussion of research problems by the Subcommittee on Research.
3. Participation in the Fatigue Conference at Purdue University on April 9, 1948. (Attendance—80)
4. Cooperation with the Administrative Committee on Simulated Service Testing and other A.S.T.M. Committees. Liaison with other societies on fatigue activities.
5. A cooperative arrangement has been made with the British Mechanics Engineering Research Board represented by Herbert J. Gough.

The present officers were reelected for the ensuing term of two years.

This report has been submitted to letter ballot of the main committee, which consists of 33 members; 27 members returned their ballots, of whom 27 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

R. E. Peterson,
Chairman.

O. J. Horger,
Secretary.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] ASTM Bulletin, No. 153, August, 1948, p. 36.

REPORT OF COMMITTEE E-11*

ON

QUALITY CONTROL OF MATERALS

Committee E-11 on Quality Control of Materials was organized in June, 1946, to promote the knowledge of statistical quality control methods and their application to specifications and methods of test. Since that time the planning of the general outlines of the committee's work and the organization of a number of task groups to carry out these plans has been completed. It is the plan of the committee to prepare a series of manuals outlining procedures and practices in areas of statistical quality control having a direct relation to A.S.T.M. activities. Six task groups have been appointed by Committee E-11 and are working on the problems assigned.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman,H. F. Dodge.
Vice-Chairman, A. E. R. Westman.
Secretary, O. P. Beckwith.

Committee E-11 sponsored the Symposium on Usefulness and Limitations of Samples[1] held at the Annual Meeting of the Society this year. The symposium comprised the following papers:[2]

"Sampling and Its Uncertainties," by S. S. Wilks.

"On Variation in Materials, Testing, Sample Sizes," by Leslie E. Simon.

"The Amount of Inspection as a Function of Control of Quality," by G. R. Gause.

REVISION OF TENTATIVE

The committee recommends that the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 – 40 T) be revised as appended hereto.[3] The proposed revised recommended practice is based on the subject matter of Sections 3, 4, 5, and 6 of the present E 29. It is a simplification of E 29 and was prepared to be of maximum usefulness to specification-writing committees.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ORGANIZATION AND ACTIVITIES OF TASK GROUPS

Group 1 on Presentation of Data (R. F. Passano, chairman) is preparing Sections 1 and 2 of the new proposed A.S.T.M. Manual on Quality Control of Materials, such sections to comprise editorial revisions of (*1*) the main section of the A.S.T.M. Manual on Presentation of Data, and (*2*) Supplement A thereof. The content is to be substantially unchanged, except that consideration is to be given to the omission of text material on skewness. It is expected that the work of the group will be completed this year.

Group 2 on Control Chart Method of Presentation (A. E. R. Westman, chairman) is preparing Section 3 of the new proposed A.S.T.M. Manual on Quality Control of Materials, such section to comprise editorial revision of Supplement B of A.S.T.M. Manual on Presentation of Data. The content is to be substantially unchanged, except for modifications to treat separately charts for number of defects and charts for number of defectives, as covered in "Note Regarding April, 1943, Reprinting," and to include additional examples, if needed. It is expected that the work of the group will be completed this year.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Issued as a separate publication, December, 1948.

[2] See pp. 857 to 895.

[3] These revised tentative recommended practices were accepted by the Society and appear in the 1948 Supplemeents to Book of A.S.T.M. Standards, Parts I-A, I-B, II, III-A, and III-B.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Group 3 on Numerical Requirements in Standards (Karl L. Fetters, chairmna) prepared the revised Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 - 40 T) referred to earlier in this report.

Group 4 on Sampling Plans (G. H. Harnden, chairman) is making a survey of sampling plans used in A.S.T.M. standards and tentatives to disclose the variety and types of materials in such manner as to make statistical evaluation of these plans possible. It is expected that the work of the group will be completed this year.

Group 5 on Planning Interlaboratory Test Programs (Grant Wernimont, chairman) is working on the planning for the collection of multi-source data intended specifically for aiding A.S.T.M. committees. This report is to include consideration of such items as statement of the objective, utilization of prior knowledge, selection of testing equipment, number of contributing sources, number of samples from each source, method of selecting samples, forms for collecting data, and methods of statistical analyses. It is to be prepared in a condensed form suitable for use as a section of the A.S.T.M. Manual on Quality Control of Materials.

Group 6 on Sampling Fluctuations of Averages (A. G. Scroggie, chairman) is preparing a brief presentation on the distribution of averages of samples of size *n* from a known distribution. An application is the determination of the size of sample that must be taken for a desired precision of an average.

The committee has assisted other committees of the Society in working out problems covering special phases of application of statistical quality control to A.S.T.M. problems. Committee E-11 is sponsoring a Symposium on the Usefulness and Limitations of Samples, which will be held on Tuesday, June 22, at the Annual Meeting in Detroit, Mich.

This report has been submitted to letter ballot of the committee, which consists of 22 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

ISOTOPES AND THEIR APPLICATION IN THE FIELD OF INDUSTRIAL MATERIALS[1]

TWENTY-SECOND EDGAR MARBURG LECTURE

BY PAUL C. AEBERSOLD[2]

> "There is nothing which can better deserve your patronage than the promotion of science."—*George Washington* in his first annual message to Congress.

INTRODUCTION

At the outset I wish to go on record as not being an authority in the field of industrial materials—in fact I know very little about the subject. My training is in a field that only a few years ago might have been considered quite remote from industrial materials, namely, nuclear physics. As one who has spent most of his time concerned with basic science, I must confess that I had never given much consideration to the field of testing materials. Like the average person I had simply taken for granted the high quality and special standards of the materials and products of industry. With just a little reflection, however, it was obvious that within just one generation truly amazing progress has been made in the field of industrial materials and that this progress has been the result of a tremendous scientific and technical development much of which was aimed specifically at the study and improvement of materials. On further consideration it was clear that the relation between materials and nuclear physics is more closely knit than one might offhand expect—developments in each field appreciably affecting the other. Atomic energy, which is largely applied nuclear physics, is an outstanding example of the reciprocal relationship of materials and basic science. Only after such reflection did I, as an outsider to the field of engineering materials, become bold enough to accept the privilege of presenting your annual Edgar Marburg Lecture.

The average person may not be sufficiently aware of the great part that industry and industrial materials played in the spectacular success of the atomic energy project. Yet, this astounding success is striking evidence of the great combination of creative and productive power that derives from close teamwork between science and technology—between science and industry.

Also the average person may not fully appreciate that advances in atomic energy, in industry, and in fact in all fields of application, have been possible only because of the rapidly acclerating advance in fundamental and applied science. In all fields, fundamental research is the indispensable key to future advance. Results of basic research are the forerunner of all technical and practical application.

It should be realized that research and

---

[1] Read on June 24, 1948, before the Fifty-first Annual Meeting of the American Society for Testing Materials, Detroit, Mich.

[2] Chief of the Isotopes Division, United States Atomic Energy Commission, Oak Ridge, Tenn.

production go hand in hand because they derive from basic urges in man. Man is driven by the innate urges to *know* and to *create*. These have set him upon a never ending course to *investigate* and to *construct* and *produce*. First, he has felt urged to investigate the universe and all the materials in it. Next, he has taken the knowledge and the materials and has put them together in new ways to create new things to satisfy his wants, his comfort, and sometimes just his fancy. The urge to know is the basis of fundamental science. The urge to create is the foundation of technology, and of industry.

These two—science and industry—are mutually dependent for progress and support and are stimulating to each other. As science progresses so does technology. As technology progresses so does its practical application in medicine, agriculture and industry; in the output of food and consumer goods; in the alleviation of disease, discomfort and material want in human society. In the last two centuries there have been countless demonstrations that science is a great benefactor of man's material needs. Time does not permit us to consider here the broad and critical problem of whether man's moral and spiritual growth can keep apace of the material, but we will take faith that it can.

The most spectacular scientific advance in recent years has come from the increase in knowledge concerned with the basic transformations of matter and energy—of matter into other matter and of matter into energy—that is, in knowledge and application of nuclear energy, the so-called "atomic energy." A whole new technology—and indeed a new industry—is flowing from this rapidly expanding field of science.

First impressions are often lasting impressions. With regard to atomic energy this is unfortunate, since the early military exploitation of the tremendous power of atomic energy has overshadowed its capacity for peaceful applications and consequent rich rewards to human welfare.

Now that atomic energy is being turned more and more toward peacetime applications, the horizons and opportunities for industry in this new field have become greatly broadened. Let us in the short time available here be as concrete as possible about these applications.

As the great tree of atomic energy matures it will bear the fruit of rich peacetime benefits for industry—for science, medicine, and agriculture as well—for the over-all national welfare and strength. These benefits may be derived from at least four broad phases of development as follows:

1. Application of results of researches in atomic science to other fields, as for example, the development of special materials and equipment of use to industry in general,
2. Use of atomic energy to produce heat and electrical power,
3. Use of atomic radiation and atomic heat to induce chemical and physical change, and
4. Applications of special radioactive and stable forms of elements, or isotopes, for tracing atoms and for other unique purposes.

Full treatment of each of these phases would require long discussion. Volumes could be written just on the subject of power. Hence, it is only possible here to discuss some of these items briefly. I will, however, try to make as concrete and useful a picture as possible.

GENERAL RESEARCH DIVIDENDS

First, let us look at some of the likely industrial benefits which may be derived from the development of atomic energy but which at the same time are not directly a phase of atomic energy or its products. I am speaking of the advancements made in the development of special materials, equipment, and instrumentation.

The materials production and testing program, a necessary prerequisite for the construction of the various atomic energy plants, was a tremendous operation. Design specifications for each of the plants—the nuclear reactors at Hanford, Washington, and the electromagnetic and gaseous diffusion separation plants at Oak Ridge, Tennessee—called for fantastic quantities of high-grade materials. For instance, the first prerequisite in reactor (pile) design is absolute purity of materials. The chain reaction which is the basis for reactor operation can be maintained only if efficient use is made of the neutrons released during fission of the uranium 235. To produce plutonium, which was the wartime objective of reactor operation, this efficiency has to be stepped up to a point where there are enough neutrons not only to sustain the chain reaction but also for converting uranium 238 to neptunium, the parent of plutonium. Impurities in both the uranium and the graphite used as the moderator tend to capture these neutrons; thus the impurities must be held to a minimum.

The production of highly pure uranium was only one phase of the materials development program. Comparable programs were carried out for other materials such as graphite, thorium, beryllium, and aluminum. High-purity grades of these materials may prove useful in other industrial installations. The lessons learned and the data acquired during these development programs will certainly be valuable information for future applications.

As a striking example of the way in which research on materials may suddenly assume unexpected significance, it is interesting to recount a research study being made on uranium metal (**1**).[3] Prior to the war a large electrical products concern was carrying on a research program designed to investigate the possibility of using high-purity uranium metal as a substitute for tungsten for the filaments of lamps. The "know how" gained on the properties and purification of uranium was of immeasurable assistance in speeding up the initial production of uranium metal for the atomic energy project.

Thus far we have mentioned only those materials pertaining to nuclear reactor design. The materials developed during construction of the uranium 235 separation installations may possibly be of more immediate interest to other industries. Perhaps the most interesting of these are the fluorocarbons, different species of which now number in the thousands (**2, 3**). These materials, never before synthesized even on a laboratory scale, were developed to compensate for a plant-scale corrosion problem. In consistency they range from light oils to heavy tars to plastics and in applications from lubricants to valve seats. Some of these materials are now available on the open market and, as stated before, they are particularly noted for their corrosion resistant characteristics. Another material which "reached maturity" as a result of Project interest is gaseous fluorine (**4, 5, 6, 7**). Formerly synthesized on laboratory scale only and regarded as a "bad actor" by research chemists, elemental fluorine is now produced on plant scale and shipped like other industrial chemicals in cylinder lots. Because fluorine is the most chemically reactive material known, many engineers believe that its use will become widespread throughout industry.

Industry also stands to gain in other areas, outside of materials. For instance, instruments, especially of the pneumatic and electronic type, designed for plant operation by remote control, are certainly subject to alteration and adaptation to other manufacturing plants. The case is comparable for heavy equipment such as blowers and pumps. Of course, industry has already benefited to some degree from the specialized training received by its engineers and from access to declassified technical publications. The largest scale benefits, however, will come from the products and by-products of atomic energy.

---

[3] The boldface numbers in parenthesis refer to the list of references appended to this paper, p. 522.

USEFUL ATOMIC POWER

Now let us turn to the power aspects of atomic energy or, more specifically, the possibility of using atomic energy to produce heat and electrical power (**8, 9, 10**).

Atomic energy devices depend on the great energy of nuclear fission—the splitting of the nuclei of certain heavy atoms. Fission of a uranium 235 atom, for example, releases about three million times more energy than any chemical reaction of an atom—three million times more, say, than the energy released in the oxidation of a carbon atom, as in the burning of coal or oil. In a ton of natural uranium, there are approximately 14 lb. of the readily fissionable uranium isotope, U 235. The latent energy in this amount of fissionable material is roughly 154,000,000 kw. hr.—equivalent to the energy supplied by the burning of 21,000 tons of coal.

This energy is not being utilized in the piles which are in operation today because they were purposely designed for the production of the fissionable material, plutonium, and it was simplest to design them to operate at low temperatures. Present reactors operate at less then 100 C. if cooled by water and 150 C. if cooled by air. In any nuclear reactor, the temperature at which the power is generated must be controlled because of limiting factors such as the melting of the structural materials. Theoretically, however, the limit of the temperature attainable with fission is of the order of billions of degrees, since this is the thermal equivalent of the energy at which the fission fragments fly apart.

The problem of power pile development, therefore, resolves itself largely into a problem of materials testing and development. Not only is it necessary to find materials which will stand up under high temperatures, but it is also necessary that these same materials withstand nuclear radiations and certain types of chemical corrosion. Some of these materials will also need to have excellent heat-transfer properties. When such materials have been developed, one of the major obstacles to power pile development will have been hurdled.

It is appropriate here to note some of the other problems associated with power pile development. There is for instance, a limit to the size of a reactor—a size below which a chain reaction will not take place. This is known as the "critical size." In order to sustain a chain reaction more than one neutron must be liberated in fission for each one captured. The larger the reacting unit is made, the less chance there is for neutrons to escape to the exterior and be lost for sustaining the chain reaction. The critical size is smaller when using enriched uranium or pure fissionable materials than when using ordinary uranium.

Actually the size of the complete pile structure is considerably greater than the critical size of the reacting unit or "fuel box." This is necessary because heavy radiation shielding is required to protect operating personnel. The radiation from the reacting unit may be greater than that from many tons of radium, and the concrete shield surrounding the unit may be several to many feet thick.

It is not possible here to give accurate dimensions, but we can give indications of over-all size. We do know that the space occupied by a 10,000 to 50,000 kw. nuclear power reactor will be considerably less than that occupied by a coal-steam plant generating the same power. We do know that the size of any practical unit will not be sufficiently small to place in your basement for heating purposes or under the hood of your automobile. The rôle of the atomic power plants of tomorrow will be that of supplying heat or electrical or mechanical power on a large scale.

The heat derived from the reactors will no doubt be valuable for large-scale space heating and for commercial processes of heating, drying, etc. Reactors will be used to heat steam or other gas for operating turbines, or for other mechanisms operating at high temperatures and pressures. No doubt nuclear reactors can be developed as ideal power units for ships. They will be compact—space will not be required for the storage of coal—and it will not be necessary to make stops and detours in order to refuel. One advantage for a possible submarine application is that atomic fuel does not require oxygen.

A nuclear power plant would undoubtedly have certain advantages over present conventional power plants. A few of the possible advantages are:

1. To serve the power needs of localities where the cost of oil, coal or water power is uneconomical, particularly those localities which otherwise have rich industrial aspects.

2. To provide compact power for large size mobile units, such as ocean liners, warships, submarines, and possibly large airplanes.

3. To make it possible to decentralize industrial regions. Industries tend to group together where cheap power is available.

4. To produce radioactive materials or radioisotopes. Radioisotopes are produced by fission and by the irradiation of material placed in the pile.

Power derived from atomic energy is expected eventually to cost about the same as power now derived from other forms of energy. Present indications are that atomic power will supplement rather than compete with other forms of power.

Solution of all the inter-related and complex problems of conservation of our atomic resources, as well as of fitting atomic power into our society and industry, will require the combined effort of economists, metallurgists, chemists, miners, physicists, and administrators. The working out of the details of a national and world atomic power program will extend over the span of several generations—and longer. We must not, however, forget the many years—about eighty years—it has taken for the oil industry to reach its present highly developed status in efficient utilization of our energy resources. And, while oil can be converted to energy by simple combustion, the conversion of atomic fuel to energy is much more of a technical problem.

We should be able, however, to witness the performance of a working model of a production-type nuclear power reactor within the next ten years. Probably one of the most authoritative evaluations of the timetable of atomic power is the recent statement of the General Advisory Committee of the United States Atomic Energy Commission, as follows:

"Assuming even a most favorable and rapid technical development along these lines a word of caution is needed as to time scale. We do not see how it would be possible under the most favorable circumstances to have any considerable portion of the present power supply of the world derived from nuclear fuel before the expiration of 20 years."

INDUCTION OF CHEMICAL AND PHYSICAL EFFECTS

A nuclear reactor is unique in two capacities: (*1*) ability to supply large quantities of heat at temperatures higher than those of usual energy sources, and (*2*) production of nuclear radiations, the neutrons and gamma rays, in fantastic intensities. These capacities may find special industrial applications.

The extra high temperature heat may prove valuable for inducing certain chemical reactions to take place more efficiently than they do at the temperatures now economically or feasibly attainable for commercial scale operations. For example, at sufficiently high temperatures the commonly available gases nitrogen and methane will react to form hydrogen cyanide, a very important commercial product. It is possible that chemical reactions of this type could be carried out cheaply on a large scale with extra high temperature heat from a nuclear reactor.

The very intense nuclear radiations from a reactor can also be used to produce chemical and physical changes in materials. Most of these changes are detrimental to the normally useful properties of the material. It is possible, however, that some of the induced changes may give the material unique properties which will be applicable for special utilizations. The usefulness of radiation for industrial processing is a question that will only be answered after a great deal of further development—one of those unpredictable things of research.

ISOTOPES

Now we come to one of the fruits of atomic energy which has "ripened and is ready for market"—the applications of radioactive and stable isotopes. These applications are already varied and widespread and will no doubt continue to grow in number and importance (**11, 12, 13**).

The word isotope is derived from two Greek words "iso" and "topos," meaning same and place, and this very correctly gives it technical connotation. An isotope is defined as one of two or more species of an element, occupying the same position in the periodic table, identical in chemical behavior, and distinguishable only by small differences in atomic weight or by radioactive transformations. For example, carbon atoms are found naturally with atomic weights 12 and 13 and can be produced artificially with weights 10, 11 and 14; these are all isotopes of carbon. Isotopes undergoing radioactive transformations are known as radioactive isotopes or simply "radioisotopes."

The radioactive species of isotopes occur in only limited number in nature—almost entirely in the elements heavier than lead, familiar examples being radium, thorium, and uranium. However, even before the war, nuclear scientists were successful in their attempts to synthesize radioactive isotopes of nearly all the elements by bombarding normally stable elements with sub-atomic particles in "atom-smashing" devices such as the cyclotron (**14**). The radioisotopes produced in this way were extremely small in quantity, and in general too costly to be available to many investigators.

The wartime development of the chain-reacting atomic pile or reactor revolutionized the production of radioisotopes. Production of most of the isotopes has been increased by thousands to many millions of times that possible with the cyclotron. These "man-made atoms" are now being used for research and application by about 500 groups in hospitals, industrial companies, universities, research centers, medical clinics, agricultural experiment stations, and other institutions all over the United States. More than forty industrial research groups alone are now using radioisotopes in about one hundred individual research and development projects. Indeed, isotope production by the pile at Oak Ridge, Tenn. is not only sufficient to meet our domestic needs but to permit investigators in a score of foreign countries to receive shipments for medical therapy and fundamental research.

The fact that radioisotopes of most of the elements can now be made in the pile makes possible a wide scope of use in such fields as medical therapy, animal and plant physiology, bacteriology, physics, chemistry, industrial research and control, and metallurgy (**15, 16, 17, 18, 19**). Further, the expense of production no longer stands as a barrier to the would-be user. A striking example is the case of radioactive carbon, carbon 14, a radioisotope with almost unlimited usefulness in organic chemistry and biology. It has been estimated that one millicurie of C 14 produced in the cyclotron would cost $1,000,000, whereas the same amount of pile-produced carbon 14 is now available at a cost of only $50, and it can probably be produced even more cheaply.

The great usefulness of isotopes arises from two facts: First, the radioactive isotope will exhibit the same chemical behavior as the stable species or stable isotopes of the element. Second, the radioactive isotopes emit radiations which will determine their identity and location. Because of these two properties it is possible to use radioisotopes as tracer atoms—atoms which will follow along exactly with the usual species of atoms, but which will "give away" where they go even in the most complicated systems and processes. Tracer atoms are one of the most powerful tools yet placed at the disposal of scientific investigators.

The rôle of these radioactive materials in industrial research, already apparent on the basis of preliminary reports, is potentially great. They are being used as tracer atoms in research programs covering such a scope of topics as friction, lubrication, diffusion, steelmaking, vulcanization, polymerization, corrosion, catalysis, well logging, organic synthesis, etc. (**20, 21, 22, 23**).

Isotope Properties:

To appreciate how isotopes can be applied, one is required to have at least a working knowledge of their properties, preparation, and limitations. It is therefore appropriate to backtrack slightly and approach the subject from a more technical viewpoint.

As pointed out earlier, isotopes are atoms—specific species of atoms. The stable species, or isotopes, of a given element are distinguishable by small differences in atomic weight. The radioactive isotopes of the same element are also distinguishable by radioactive transformations. The nuclear physicist thinks of isotopes as groups of neutrons and protons ("nucleons") with the same atomic number but different atomic weights. Associated with each stable nucleus is a definite number of nucleons arranged in a configuration having a stable energy balance. For the radioactive nucleus, however, the energy balance is not stable and, through nuclear phenomena little understood, the nucleus approaches a more stable balance state by disintegration and the emission of energy in the form of particles or radiation (**24, 25**).

The radioactivity of a radioisotope "decays" over a period of time because of the disintegration of its atoms. The period of time associated with this decay may be expressed in terms of the half-life. A half-life is the length of time in which half of the activity of the isotope will have disappeared. In the next half-life period half of the remaining activity will disappear, etc. (**26**). Each radioisotope has its own characteristic half-life; as for example, the half-life of carbon 10 is 8.8 sec., whereas the half-life of carbon 14 is 5000 years.

There are three different types of radiation which radioisotopes may emit. These are alpha particles which are nuclei of helium atoms, beta particles which may be either positively or negatively charged electrons, and gamma rays or X-rays which are photons of electromagnetic radiation. Each radioisotope may emit one or more of these types of radiation, but the type of radiation and the energy of emission will always be the same for any given isotope. For instance, both carbon 14 and phosphorus 32 always decay with the emission of a negative electron but without gamma radiation. They are thus spoken of as being pure beta emitters. The beta radiation emitted by phosphorus 32 has a maximum energy value of 1.69 Mev. whereas for carbon 14 the corresponding value is only 0.154 Mev. (Mev., which is an abbreviation for "million electron volts," is defined as the energy acquired by an electron falling through a potential drop of one million volts. 1 Mev. = 1.6×10^{-6} erg = 1.18×10^{-13} ft-lb.)

The unit of measurement in the quantitative determination of radioactivity is the "curie." The curie was originally defined as the amount of radon in equilibrium with 1 g. of radium. This amount of radon decays at a rate of 3.7×10^{10} disintegrations per second. More recently the curie has arbitrarily been taken as the amount of radioactivity associated with any radioisotope which is disintegrating at the rate of 3.7×10^{10} disintegrations per second (**27**). In most radioisotope applications, however, the most frequently used units are the millicurie and microcurie, respectively equivalent to 3.7×10^{7} and 3.7×10^{4} disintegrations per second.

Isotope Production:

The production of a radioisotope involves upsetting in some way the balance of nuclear particles or nucleons in the nucleus of an element. In the cyclotron this is accomplished by accelerating some charged sub-atomic particle such as a proton or deuteron to a speed where it has sufficient kinetic energy to overcome the electrostatic repulsion of the heavily charged nucleus of the target atom, that is, to break through its "potential energy barrier," and thus to lodge itself in that nucleus. The bombarding particle must have sufficient energy not only to get itself into the target nucleus but also to force some other particle out. The energy necessary for such an operation, of course, is dependent on the type of particle going in and the type coming out.

Production of radioisotopes with a nuclear reactor or pile is quite different from cyclotron production. The bombarding particles are neutrons which have no electrical charge and which, therefore, can penetrate the potential barrier of the target nucleus with little or no energy. Energy to get a particle out of the nucleus is not necessary in many pile reactions because only gamma or electromagnetic radiation is emitted. The pile is also unique for radioisotope production in that, because of its large volume, it is possible to bombard a very large number of different lots of target materials at the same time. Production capacity is dependent on the neutron density or "flux" of the pile. Neutrons not used to sustain the chain reaction are available for irradiation of stable isotopes to form radioisotopes. In the Oak Ridge pile the chain reaction utilizes slow or "thermal" neutrons. The thermal neutron flux where most of the radioisotopes are produced is about 5×10^{11} neutrons per sq. cm. per sec.

Pile-produced radioisotopes may result from three different nuclear reactions (see Fig. 1). The simplest of these is referred to as neutron absorption where the stable target element absorbs a neutron and a quantum of gamma radiation is emitted. Since this reaction involves a "neutron in and a gamma ray out" it is called an "n, gamma" reaction. A radioisotope formed in this way is an isotope of the parent element and, therefore, cannot be chemically separated from the original target material.

A second type of pile reaction is known as transmutation, since the radioisotope produced is a different element (hence transmuted) from the target element. In the most frequent cases of transmutation the stable isotope absorbs a neutron and a proton is emitted. This is called an "n, p" reaction. Since the resulting isotope differs in elemental form from the target element, it may be chemically separated. Such chemically separated isotopes are referred to as being "carrier-free," that is without a significant quantity of stable atoms of the same element to act as "carrier."

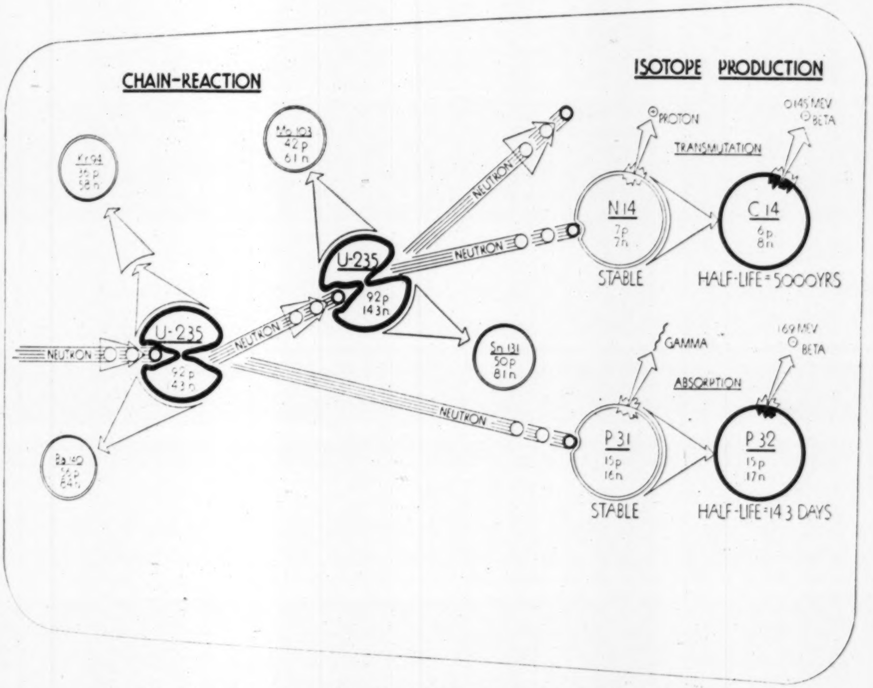

CHAIN-REACTION
ISOTOPE PRODUCTION
Kr 94
36 p
58 n
Mo 103
42 p
61 n
U-235
92 p
143 n
U-235
92 p
143 n
Sn 131
50 p
81 n
Ba 140
56 p
84 n
NEUTRON
NEUTRON
NEUTRON
NEUTRON
NEUTRON
PROTON
0.145 MEV
BETA
TRANSMUTATION
N 14
7 p
7 n
STABLE
C 14
6 p
8 n
HALF-LIFE = 5000 YRS
GAMMA
1.69 MEV
BETA
ABSORPTION
P 31
15 p
16 n
STABLE
P 32
15 p
17 n
HALF-LIFE = 14.3 DAYS

FIG. 1.—Reactions Used in Pile Production of Radioisotopes.

A third type of reaction by which radioisotopes are produced in the pile is fission. When uranium fissions, it breaks into radioactive fragments called fission products. These fragments range in atomic number from element 30 to element 64. The fission products may be separated from the uranium and subsequently separated from each other, to be used either as individual radioactive species or as groups of species. The radioisotopes most frequently used for general research and application are produced by the first two reactions.

The mechanical operation of producing radioisotopes in the pile merely involves the following sequence: stop the chain reaction, place the stable element or compounds (target material) in the pile, start the chain reaction again, allow the target material to remain for a certain period of irradiation, again stop the chain reaction, and remove the material for chemical processing and packaging. Small aluminum test tubes ($\frac{5}{8}$ in. in inside diameter by 3 in. in length) are used for retaining the material to be irradiated. Aluminum is ideal for this purpose because it not only has a low absorption for thermal neutrons, but the activity induced in it decays rapidly. The aluminum test tube containing the stable isotope is inserted into a graphite "stringer" which is then pushed into the pile. The duration of pile irradiation is dependent upon the rate of decay of the resulting radioactive isotope, the relative absorption of neutrons by the target element, the level of activity desired, and certain other variables. For those reactions producing isotopes of short half-life (say 4 days or less) the target material is usually allowed to remain in the pile for a week, whereas for radioisotopes of longer half-life the target material may remain in the pile from several weeks to several months. Depending on the nuclear reaction involved in the preparation of the radioisotope, the material may or may not be chemically processed prior to packaging and shipping. Figure 2 shows a schematic view of the Oak Ridge pile and the method of inserting materials for irradiation.

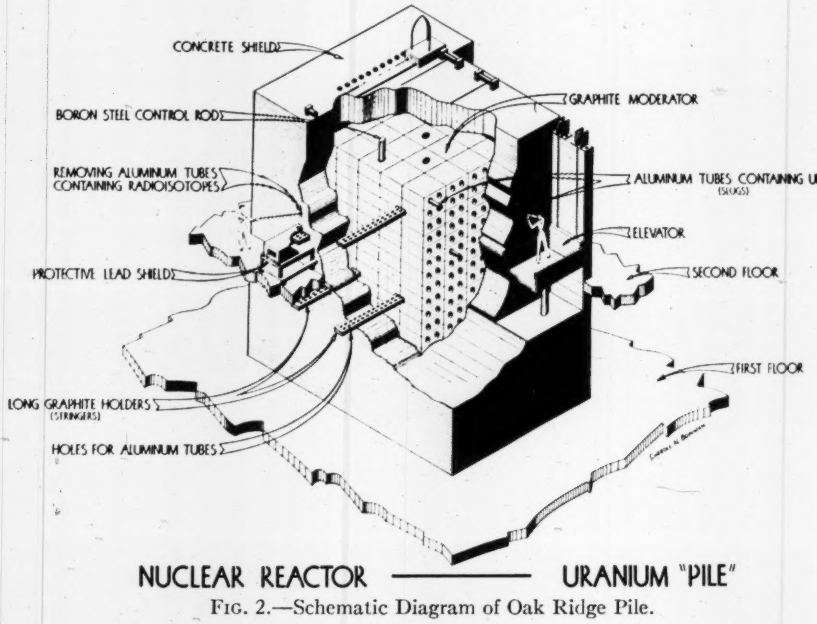

CONCRETE SHIELD
BORON STEEL CONTROL RODS
REMOVING ALUMINUM TUBES CONTAINING RADIOISOTOPES
PROTECTIVE LEAD SHIELD
LONG GRAPHITE HOLDERS (STRINGERS)
HOLES FOR ALUMINUM TUBES
GRAPHITE MODERATOR
ALUMINUM TUBES CONTAINING URANIUM (SLUGS)
ELEVATOR
SECOND FLOOR
FIRST FLOOR
NUCLEAR REACTOR ——— URANIUM "PILE"

FIG. 2.—Schematic Diagram of Oak Ridge Pile.

Radioisotope Measurement:

Determination of the amount of a radioisotope is based on a measurement of the radioactivity. When alpha, beta, or gamma rays pass through matter, they ionize the atoms in the matter, thereby producing electrically charged atoms called ions. It is these ions rather than the rays themselves that are usually detected and measured (**28, 29, 30, 31, 32, 33**).

Charge and speed are always required for the detection of nuclear particles. Radiation such as gamma rays which have no charge are detected by secondary effects which cause the formation of charged, fast moving particles.

The instruments most often used to detect and measure radiation are the electroscope, the Geiger counter, and the ionization chamber. The operation of the first is dependent on the ions, produced by the radiation, dissipating an electrical charge originally placed on the instrument. The operation of the Geiger counter is based on the production and collection of an avalanche of secondary ions to register a pulse of current. The operation of the ionization chamber is also based on the collection of ions, but in this case the total number of primary ions. The electroscope has the advantage of not requiring electronic circuits and power supply. Geiger counters and ionization chambers have the advantage of rapidity of measurement and the possibility of automatic recording of results.

Another method of measuring radioactivity is based on the fact that radiation exposure of a photographic film causes it to darken upon development. The degree or density of the darkening is proportional to the quantity and intensity of radiation. Radiography is based on the exposure of a photographic plate by the radiations from an external source (**34, 35, 36, 37, 38**). For example, in testing a weld, the radiations from a source are allowed to impinge against the weld while a photographic plate is placed on the opposite side of the test section. The degree of exposure on the plate will show the location of possible flaws in the weld. Autoradiography also depends on photographic detection techniques, but in this case, the radioactive material is actually incorporated into the material to be tested. The photograph is made by placing the test sample in contact with the film. This technique shows the distribution of active material in the test sample.

Facilities And Safety Precautions:

The radiations from radioactive materials are potentially harmful to health. To minimize the possible hazards, special facilities and safety precautions are usually required for handling and using radioisotopes (**39, 40, 41, 42, 43**). The nature of these requirements, however, is dependent on the type and level of activity being handled. For instance, in handling small quantities, say, less than 10 microcuries of a low-energy beta particle emitter such as carbon 14 (Mev. = 0.154) no special safety precautions would be necessary other than those usually taken in an experimental laboratory to prevent inhalation or ingestion of toxic materials. On the other hand, in working with a larger activity of a gamma ray emitter such as cobalt 60 much more stringent safety precautions are necessary because of the great penetrating power of the gamma radiation. In this case it is necessary to carry out all operations behind a concrete or lead shield sufficiently thick to reduce the radiation intensity to a safe level.

Certain low levels of radiation may be absorbed by the body over an extended period of time without showing any effect on the bodily processes associated with normal health. The maximum permissible exposure of X-rays or gamma radiation is measured in roentgens. The roentgen (r) is that quantity of X- or gamma radiation which falling on dry air under standard conditions produces ionization to the extent of one esu (electrostatic unit) of charge of either sign per cubic centimeter. Since the element of time is involved in radiation exposure, it is customary to measure radiation in roentgens per hour. A roentgen of X- or gamma radiation when absorbed in tissue produces about 83 ergs per gram of tissue. Since the roentgen is properly defined only for X- or gamma absorption, the term roentgen equivalent physical (rep) is employed for the equivalent rate of absorption produced by other types of radiation. The maximum permissible exposure value is usually taken as 0.1 r per 24-hr. day, although some persons favor the lower value of 0.3 r per week.

To maintain this low permissible dosage, the radioisotope user frequently measures the amount of radiation present in his working area with the aid of portable radiation meters. Also to guarantee further his personal protection, he may wear film badges or small condenser-like meters called dosimeters (see Fig. 3). The exposure to the film badge and the amount of current which has leaked off the previously charged dosimeter are directly proportional to the amount of radiation received.

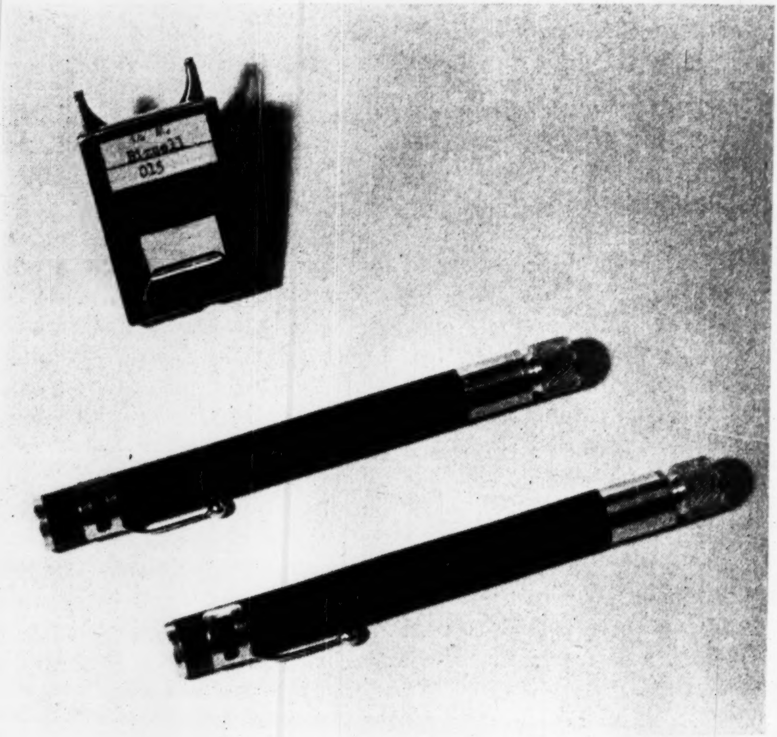


FIG. 3.—Dosimeters and Film Badge.

Facilities in a radioisotopes laboratory should be designed and spaced to afford maximum safety and minimum contamination (**44, 45, 46**). Experimental techniques, excepting those modified by safety precautions, are similar to the techniques used in quantitative work. When working with radioactive gases, dusts, or vapors, all operations should be carried out in a well-ventilated hood. When carrying out such operations as drying and evaporation, extreme care should be made to avoid spattering. Equipment coming in contact with radioactive materials may become contaminated, and, although this possibility may not constitute a health hazard, it may interfere with making accurate measurements. The same laboratory area should not be used for handling both high and low levels of activity. Quantitative measurements should not be made in the same room where radiochemical operations are performed.

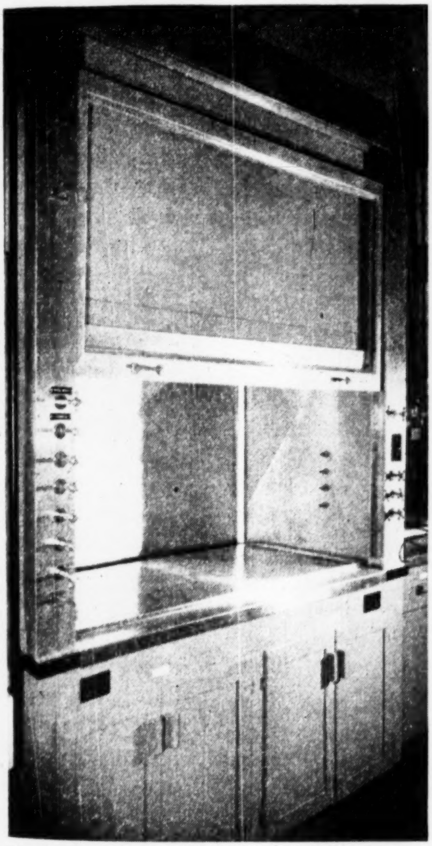


FIG. 4.—Hood Altered for Radiochemical Operations.

In many cases laboratory facilities have been altered for subsequent use in a radioisotopes laboratory. Figure 4 shows a hood which has been converted for radiochemical operations. Note that the controls for the utilities are all operated from outside the hood. Also note the stainless steel tray on the benchtop of the hood to serve as a "catch-all" in case of a spill.

The use of proper materials of construction is extremely important to laboratory design—especially the selection of those materials which may come in contact with radioactive liquids, dusts, or vapors. In working with liquids for instance it is not only necessary that all radioactive spills be "caught" in a tray or similar container but also that this tray be constructed in such a way that it has no cracks or crevices which may possibly retain some of the radioactive material after decontamination. Further, it is necessary that the tray be constructed of a material which may be easily decontaminated. To date stainless steel has been found to be one of the most satisfactory materials. Several research programs, however, are now being carried out to investigate further the problems of radioactive contamination and decontamination of various materials.

Basis for Isotope Applications:

Radioisotopes are most widely and diversely used as "tracer" atoms. In this capacity the isotope may be likened to a dynamic and revolutionary type of "atomic" microscope—one which can actually be incorporated into the process to be studied.

As tracers, radioisotopes have two unique qualities, *sensitivity* and *specificity*. Radioactivity measurements are based on the sum of a number of individual atomic events (disintegrations), whereas other chemical and physical analyses are usually based on a weight or volume determination. By using the radioisotope technique it is possible to detect the presence of extremely small amounts of material, as low as 10,000 atoms for isotopes of short half-life. If these atoms were radioactive sodium (Na 24) atoms, their total weight would be equivalent to 4×10^{-19} g. Obviously, this sensitivity is about a million million times greater than that ordinarily obtained by other methods. For radioisotopes of very long half-life a larger number of atoms are required for detection, but even for carbon 14 (5000 years) the sensitivity of detection is over a million times that of chemical methods.

The specificity of the radioisotope technique arises from the fact that it can be made to detect and follow a specific batch of atoms (the radioactive ones) independent of other atoms. A minute quantity of a radioisotope can be followed through a complicated chemical reaction or series of reactions and still be certainly identified. The complexity of processes traceable with radioisotopes is almost unlimited. The labeled atoms can be traced through a labyrinth of reactions and systems; for instance, the location of particular carbon atoms in complicated hydrocarbon molecules may be labeled, and the movement of the carbon atoms to other molecular components can then be studied.

For some applications of radioisotopes, advantage can be taken of the fact that some isotopes emit penetrating radiation which may be detected at a distance. *Detection at a distance* may permit tracer measurements to be made without interrupting or disturbing the process for the purpose of taking samples.

Radioisotopes may also be used as *sources of radiation*. In this rôle their utilizations are not unlike the applications which have been made of radium and its radioactive decay products for many years. Man-made isotopes, however, because of their many varieties can offer a much wider range of types and intensities of radiation.

Tools of Analysis:

Just as it is difficult to classify the research uses of a microscope, a balance, a colorimeter, or some other laboratory tool, so it is also difficult to classify the possible uses of radioisotopes. In the physical sciences, however, most radioisotope utilizations fall into some phase of qualitative or quantitative analysis.

The simplest type of analysis is the "yes" or "no" type of experiment where one wishes to determine the presence or absence of a particular substance in an agglomerate of other materials. By using a radioisotope to "tag" the substance, its presence or absence can be determined quickly at concentrations far below those permitted by other methods.

The real potentialities of the tracer technique, however, are employed in the more difficult type of experiment where both qualitative and quantitative measurements are required, as for example in investigating the mechanism by which a physical or chemical phenomenon takes place.

There are three distinct ways in which radioisotopes may be used as analytical tools. These may be classified as (*1*) "tracer analysis," (*2*) "isotope dilution analysis," and (*3*) "activation analysis."

Tracer analysis is the quantitative "tag" type of determination where a radioisotope in known amount is added at some stage of a process as a "label" for some one substance of the process materials. The concentration of this label (radioactivity) will accurately show the concentration of the substance in all subsequent steps of the process, even at concentrations far below those determined by standard analytical procedures.

The limitation here is the necessity for determining by some standard analytical procedure the concentration of the substance originally labeled.

Isotope dilution analysis is a modification of tracer analysis (**47, 48**). The radioisotope is mixed in known ratio with a stable isotope of the same element and added to the process materials. Any change in this ratio will be due to the dilution caused by the same element already present in the process materials. The amount of element originally present is thus determined by the amount of change in the ratio of active to stable element. This method does not require a quantitative determination of the process material at the time the radioisotope is added. Also, the ratio determination may be made on an aliquot sample. The accuracy of this method depends considerably on the degree of dilution which takes place.

Activation analysis involves the irradiation of an unknown sample in the pile and the subsequent identification of the radioisotopes produced by means of their specific radiation characteristics. In this way the elements which have been activated may be determined. This type of analysis is particularly useful where the concentration of the unknown element is too low to be identified by chemical or spectroscopic methods or where standard methods of analyses are not satisfactory because of interfering contaminants. The obvious limitations to this procedure are: (*1*) a pile or other nuclear bombarding device is necessary for irradiating the unknown sample, (*2*) the element to be analyzed may not have a suitable radioisotope for measurement, and (*3*) several elements in the material may be activated which have radioisotopes of such similar radiation characteristics as to prevent differentiation of the desired element.

Illustrations of Applications of Radioisotopes:

No amount of explanatory information is complete without illustrations. Accordingly, the following examples of radioisotope technique in industrial research problems are cited. For convenience of review we can place them in five categories: (*1*) organic chemistry and catalysis, (*2*) physical chemistry and metallurgy, (*3*) mining, (*4*) miscellaneous, and (*5*) process control.

Organic Chemistry and Catalysis.—Most of the radioisotope applications made to date in the fields of organic chemistry and catalysis have been in studying the mechanisms of organic reactions such as the oxidation and isomerization of hydrocarbons, the polymerization and vulcanization of rubber, and the dehydrogenation and aromatization of petroleum. Some research programs are being devoted solely to studies of typical organic reactions with the hope that by determining the mechanisms of these reactions, a better understanding of the general chemistry of hydrocarbons will be obtained. Investigations are of course being made of other than the reaction mechanism type, such as determining ionic mobility in hydrocarbons, studying the rôle and efficiencies of cracking catalysts, and determining the settling rates of additives, such as rust inhibitors in oil.

One interesting investigation in applied research which involves both the study of organic reaction mechanism and catalysis is concerned with the synthetic production of gasoline and other organic products by the Fischer-Tropsch process (**49**). This investigation takes on added significance when we consider the fact that gasoline is one of the most widely used materials and that some estimates of our present petroleum reserves are quite pessimistic. The Fischer-Tropsch synthesis involves the oxidation of natural gas with high purity oxygen at a pressure of 300 psi. and at temperatures from 1480 C. to 1540 C. to produce a mixture of carbon monoxide and hydrogen. This same gas mixture may also be produced by burning coal with steam and oxygen. The hydrogen-carbon monoxide mixture is then passed over a catalyst, usually iron, to produce gasoline, diesel oil, and mixtures of oxidized compounds from which alcohols, aldehydes, acids, and ketones are recovered.

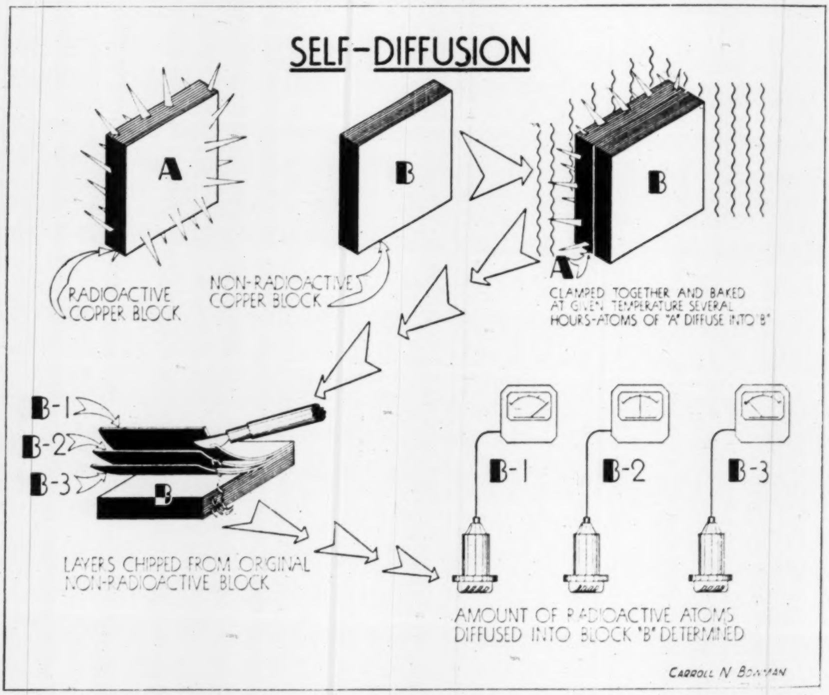
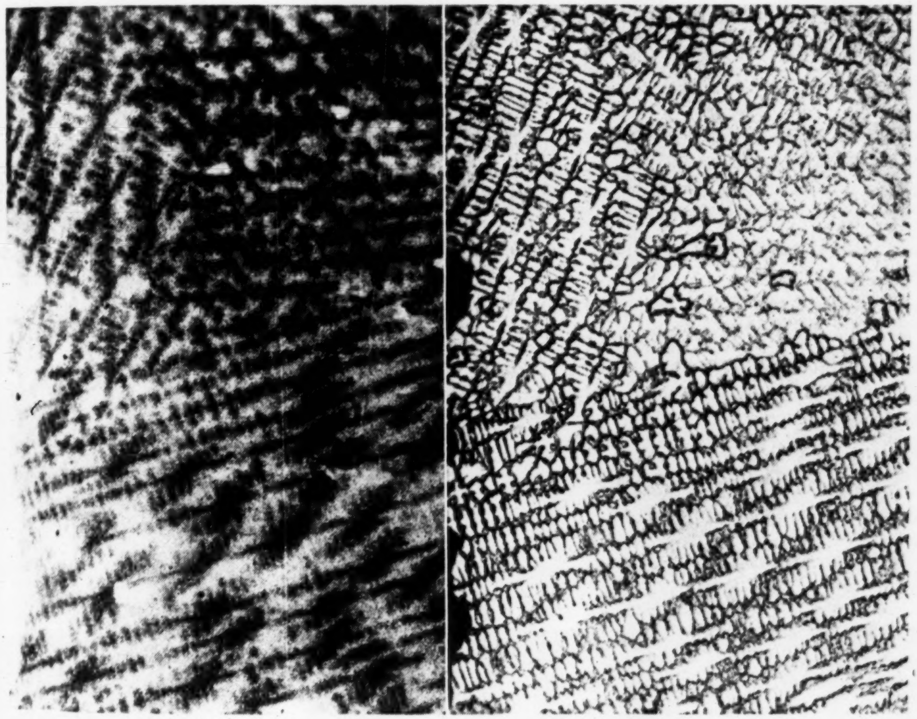

SELF-DIFFUSION
A
B
B
A
RADIOACTIVE COPPER BLOCK
NON-RADIOACTIVE COPPER BLOCK
CLAMPED TOGETHER AND BAKED AT GIVEN TEMPERATURE SEVERAL HOURS-ATOMS OF "A" DIFFUSE INTO "B"
B-1
B-2
B-3
B
LAYERS CHIPPED FROM ORIGINAL NON-RADIOACTIVE BLOCK
B-1
B-2
B-3
AMOUNT OF RADIOACTIVE ATOMS DIFFUSED INTO BLOCK "B" DETERMINED
CARROLL N BOWMAN

FIG. 5.—Self-Diffusion.

As the Germans practiced the Fischer-Tropsch process, it produced a very inferior grade of gasoline, which could be improved to adequate quality only by a large sacrifice in yield. American technologists have improved the synthesis, but there is still much to be learned about the mechanism of the reactions. For example, there has been some evidence for believing that one of the iron carbides is an intermediate in the reactions; other evidence has strongly refuted this suggestion. Recently it was reported by one laboratory on the basis of tracer studies made with carbon 14 together with thermodynamic data that iron carbide cannot be an intermediate under the conditions of temperature and pressure usually employed in the synthesis (50). Additional information of this and other types obtained through tracer studies will undoubtedly lead to a better understanding of the most desirable conditions relative to pressure, temperature, and catalyst.

Another material which has come under closer scrutiny as a result of the radioisotope technique is rubber. The processes of polymerization and vulcanization for instance have been successfully used for many years but entirely on an empirical basis. Little or nothing has been known about the mechanism of the reactions underlying the processes. It is known that vulcanization can be effected with elemental sulfur but that organic compounds containing sulfur, either alone or in conjunction with elemental sulfur, are much more effective. It is also known that in some way sulfur ties neighboring rubber molecules together, but whether this is by a —S—, —S—S—, direct —C—C—, or more complicated bridge is not known. This problem is being studied by reacting rubber with sulfur containing the radioisotope sulfur 35 and subsequently determining the location of the sulfur 35. A similar approach is being used to study the mechanism of polymerization with sulfur-bearing organic modifiers.

Physical Chemistry and Metallurgy.—Most of the metallurgical applications of radioisotopes made thus far fall into the study of steel-making reactions, such as determining the mechanism and kinetics of slag-metal reactions, oxidation and crystallization, the molecular constitution of slag and metal baths, the diffusion coefficients of metals, the metallurgical activity of one element as influenced by the presence of other elements, and the concentration gradients in solid solutions (**51, 52, 53, 54, 55, 56, 57, 58, 59**). Also in the area of metallurgy are radioisotope studies of friction and wear. The applications in physical chemistry are more heterogenous, consisting of such investigations as the migration of metallic ions into insulating compounds, the mechanism of adsorption



(*a*) Autoradiograph. (*b*) Photomicrograph.

FIG. 6—Autoradiograph and Photomicrograph of the Same Surface of a Nickel, Chromium, Tungsten Alloy with Tungsten 185.

and corrosion, and the mechanism of surface wetting and detergency.

The radioisotope technique is found to be extremely useful in investigating certain metallurgical reactions not only because of its sensitivity but also because of its specificity. An excellent example of such a case is the study of self-diffusion (**60**). As shown schematically in Fig. 5, it is possible with the use of radioisotopes to test by experimentation the theories of self-diffusion in solid metals, especially for the case of metals under stress at elevated temperatures. Two blocks of the same metal, one radioactive and the other stable, may be clamped together and subjected to a heat treatment. Following such treatment the extent to which the radioactive atoms have diffused into the stable block can be measured.

Using radioisotopes it is possible to locate a particular constituent of a solid solution and then to follow its movement when the metal is subjected to various types of treatment (**61, 62**). For instance, by using the radioisotope tungsten 185 it has been possible to determine the location of tungsten in a 70 per cent nickel, 25 per cent chromium, 5 per cent tungsten alloy. An autoradiograph of such a sample (Fig. 6(*a*)), used in conjunction with a photomicrograph (Fig. 6(*b*)), shows that the tungsten is located in the dendrites. In a cobalt-base alloy, however, it can be shown that the tungsten is generally associated with the intermetallic compounds and solid solutions between the dendrites. To date this type of study has only been possible in cases of relatively coarse structures, but it does give information which cannot be obtained with other methods of analysis, and it is probable that the resolution of this method will be improved with further application.

Although *friction and wear* phenomena are usually associated with metallurgy, they are sufficiently important to all materials to warrant separate consideration. Friction studies are not new, but, prior to the radioisotope technique, methods of analyses were not sufficiently sensitive or accurate to give an insight into the mechanism of friction (**63, 64**). The radioisotope technique is reported to be ten thousand times as sensitive for measuring material transfer as other methods, such as the taper-sectioning method, the electrographic method, and the X-ray photographic method. It also permits material transfer measurements between identical surfaces. In addition, the radioisotope technique permits "distribution" studies, that is, the detection of areas of greatest wear. Autoradiography is particularly useful in this type of measurement.

There is much controversy over the nature of friction force. To date, certain assumptions have been made such as: (*1*) the greater part of the work of the friction force is transformed into thermal energy, (*2*) part of the frictional work is stored in the form of cold work, and (*3*) work is consumed in tearing metal particles out of the sliding bodies, thus increasing the surface energy. It may be possible with a much more sensitive method of measuring the transfer of material that the basic principles underlying friction phenomena can be established.

The applied type of research along these same lines has many more immediately tangible implications. With a method of analysis sensitive enough to detect material transfer between two lubricated surfaces and specific enough to detect the areas of transfer, it should be possible to get a better understanding of such phenomena as galling and seizure in journal bearings and the running-in of piston rings and cylinders. This information of course should eventually lead to a better performance of various types of engines. As a modification of this type of investigation, tests for wear of critical engine parts without the necessity of overhaul may be developed by looking for radiotracers in the lubricating medium. Figure 7 shows a schematic diagram of radioisotope applications in friction studies.

Mining.—The petroleum industry for a long time has been using natural radioelements in well-logging operations to determine the depth and thickness of various types of strata (**65, 67, 68, 69, 70**). These data are obtained by simultaneously lowering into the well a radioactive source and a partially shielded detection instrument. Measurements are based on the amount of radiation reflected by the walls of the well and picked up by the detection instrument. Different types of strata exhibit different absorption and reflection characteristics for neutrons and gamma rays, the two types of radiation most often used for well logging.

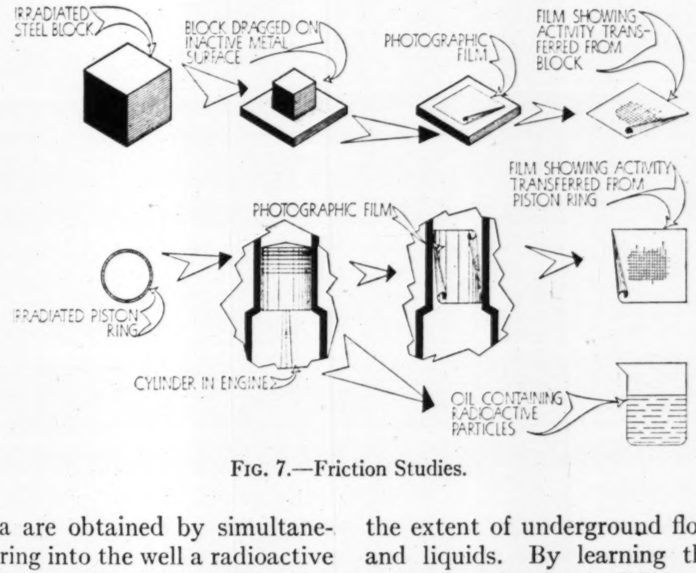

IRRADIATED STEEL BLOCK
BLOCK DRAGGED ON INACTIVE METAL SURFACE
PHOTOGRAPHIC FILM
FILM SHOWING ACTIVITY TRANSFERRED FROM BLOCK
FILM SHOWING ACTIVITY TRANSFERRED FROM PISTON RING
PHOTOGRAPHIC FILM
IRRADIATED PISTON RING
CYLINDER IN ENGINE
OIL CONTAINING RADIOACTIVE PARTICLES

FIG. 7.—Friction Studies.

Synthetic radioactive materials will probably be more versatile in their application in well logging. Studies are being made on core samples using such isotopes as radioiodine, radiochlorine, radiocobalt, and radiocalcium to determine such things as the permeability of various strata, mechanisms of flow and fluid displacement in porous media, influence of hydrated clays present in oil sands, and injection water profiles. Consideration has also been given to incorporating radioactive tracers in a large volume of gas or fluid to be injected into an oil well. A subsequent search for the tracer in samples drawn from neighboring wells will help to determine the extent of underground flow of gases and liquids. By learning the various underground routes in different parts of a field, the most efficient drilling procedures can be determined for that particular field.

Radioisotopes are also being used in such mining problems as determining the mechanism of flotation of minerals (**71, 72**). Flotation is based on heterogeneous adsorption at low concentrations (10^{-8} to 10^{-10} mol per ml.) and involves competition between various adsorbates. Also, heterogeneous exchange between adsorbed and dissolved phase is an important aspect. As in studying the mechanisms of adhesion and surface wetting, the radioisotope technique, because of its great sensitivity, is extremely useful. Since monolayer mechanisms are involved, the quantities of material transferred must be measured in parts per one hundred million. This is beyond the range of ordinary chemical analysis.

Miscellaneous Applications.—Certain industrial radioisotope utilizations do not fall into any of the categories previously mentioned and thus we will classify them as miscellaneous. Included in this category are studies on the hardening of evaporated mirrors by strong oxidants, efficiency of mixing graphite and liquids, the amount of metallic silver produced in photographic emulsion, the tarnish resistance of silver plate, and the transfer of printing ink from printing plate to paper stock.

In the latter case, for instance, a radioisotope is incorporated in a pigment or varnish which is then used in the preparation of printing inks. This radioactive ink is used to study its transfer from a printing plate to paper stock as a function of the press variables, printing ink variables, and paper variables. The radioisotope technique is found to be much more satisfactory than other methods which tend to be long and tedious and also require destroying the print.

Another radioisotope application which might fall into the miscellaneous grouping is dissipation of electrostatic charge on heavy machinery. This utilization employs the radioisotope as a source of radiation rather than as a tracer. By placing radioactive materials near the moving parts of the machinery it is possible to ionize the air surrounding the parts and thus prevent the accumulation of electrostatic charge. The radioactive material must be placed in a location to accomplish its purpose without subjecting the machine operator to the radiation. Alpha particle emitters, because of their relatively higher ionizing intensity, are preferable for such utilizations, although beta particles emitters have been used.

Process Control.—Thus far most of the applications of radioisotopes in process control (**73**) have been based on using the isotope as an external source of radiation, such as in thickness and height gages. Radioisotope tracer utilizations can however be adapted to process control. Two limitations arise in using radioisotopes in process materials: (*1*) in preventing an excessive amount of radioactivity from remaining in the finished product, and (*2*) in keeping radiation exposure of plant personnel to safe levels.

The radioactive thickness gage is dependent on the intensity of radiation transmitted through the film or sheet of material being measured. The height or level gage on the other hand is dependent on the difference in radiation transmitted through a liquid and air or on changes in the source to detector distance.

A thickness gage to measure thin rubber or plastic films, approximately 0.001 in. in thickness, has been developed by a leading rubber company, using carbon 14, a pure beta particle emitter, as the radiation source. Mechanical gages usually used in making such measurements are not satisfactory because small changes in the pressure applied to the gage contacts result in false readings. The greatest objection, however, to using a mechanical gage on a production line is that to make the measurement either the film must be stopped or a piece must be cut from the sheet. In using the radioactive thickness gage no mechanical contact with the film is required. This gage has been developed to read with an accuracy of 0.00001 in., and it is hoped that the accuracy can be increased to 0.000001 in.

Other companies are developing similar thickness gages, using different radioisotopes with more penetrating radiation, which will be adaptable to measuring thicker sheets of other materials, such as metal, glass, etc (**74**). A possible refinement for such a gage is an electronic circuit whereby the rollers for forming the sheet will be controlled by a signal from the radiation detection instrument. A schematic diagram of a radioactive thickness gage is given in Fig. 8.

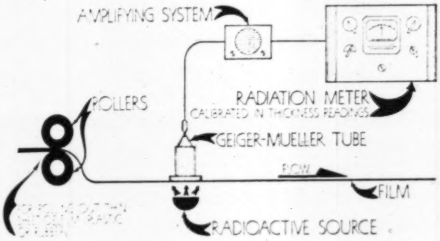

AMPLIFYING SYSTEM
ROLLERS
RADIATION METER
CALIBRATED IN THICKNESS READINGS
GEIGER-MUELLER TUBE
FLOW
FILM
RADIOACTIVE SOURCE

FIG. 8.—Radioactive Thickness Gage.

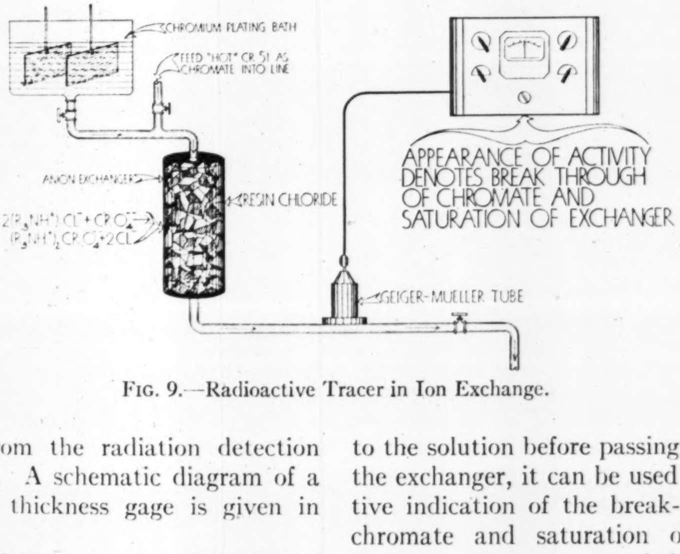

CHROMIUM PLATING BATH
FEED "HOT" CR 51 AS CHROMATE INTO LINE
ANION EXCHANGER
RESIN CHLORIDE
2(R₃NH⁺) CL⁻ + CR O₄⁼ →
(R₃NH⁺)₂ CR O₄ + 2 CL⁻
APPEARANCE OF ACTIVITY DENOTES BREAK THROUGH OF CHROMATE AND SATURATION OF EXCHANGER
GEIGER-MUELLER TUBE

FIG. 9.—Radioactive Tracer in Ion Exchange.

In the height gage or liquid-level gage the radioisotope may be contained in a float which rides on the surface of the liquid or is fixed in a stationary position with reference to the height of the liquid container. The intensity of radiation passing through the liquid and passing through air will be sufficiently different to be easily detected. One company is using a radioactive height gage to measure the height of melt in a cupola. Similar devices controlled by movement of radiomaterial and action of a radiation detection meter could be useful in many liquid flow processes.

As an example of a radioisotope tracer application in industrial processing, reference is made to Fig. 9. It has been found that after using a chromium plating bath for an extended period of time it becomes necessary to renew it.

It is not economically feasible, however, to discard the "old" bath because of its chromium content. The chromium is accordingly removed from the chromate solution by an ion exchanger. If a radioisotope, say chromium 51, is added to the solution before passing it through the exchanger, it can be used as a sensitive indication of the break-through of chromate and saturation of the exchanger. The presence of the radioisotope is also useful in the subsequent step where the chromium is recovered from the ion exchanger. A further possible development of this scheme would be to use a signal from the radiation detection instrument to control a valve which would direct the chromate solution into a second ion exchange column when the ion exchanger in the first column had been exhausted.

An application which might be considered as more closely representing a process control operation is the control of phosphorus during steel manufacture (**75**). As indicated in Fig. 10, a major portion of the phosphorus content is removed with the basic slag. If radioactive phosphorus 32 is added at an early stage of the process, the residual phosphorus in the melt following the acid slag removal may be easily and quickly determined by a radiation measurement. Because of the short half-life of phosphorus 32 (14.3 days), any activity remaining in the melt would have disappeared by the time the steel had been fabricated and was ready for market. The advantages of this method as compared to standard methods of analysis are saving of time and ease of analysis.

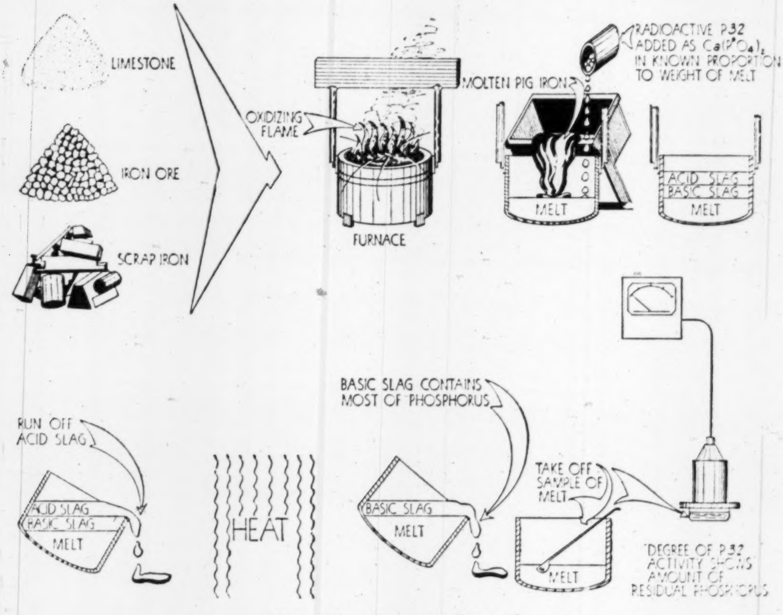

LIMESTONE
IRON ORE
SCRAP IRON
OXIDIZING FLAME
FURNACE
MOLTEN PIG IRON
RADIOACTIVE P32 ADDED AS $Ca(P^*O_4)_2$ IN KNOWN PROPORTION TO WEIGHT OF MELT
MELT
ACID SLAG
BASIC SLAG
MELT
RUN OFF ACID SLAG
ACID SLAG
BASIC SLAG
MELT
HEAT
BASIC SLAG CONTAINS MOST OF PHOSPHORUS
BASIC SLAG
MELT
TAKE OFF SAMPLE OF MELT
MELT
DEGREE OF P32 ACTIVITY SHOWS AMOUNT OF RESIDUAL PHOSPHORUS

FIG. 10.—Process Control.

A discussion of the uses of radioisotopes in process or quality control would not be complete without mention of their application in radiography. Several industrial companies are using radioisotope sources, usually gamma ray emitters such as cobalt 60, in the radiographic examination of welds and cast parts. Artificially produced radioisotopes have the advantage over natural radioelements in offering a wider selection of energies of gamma radiation.

Stable Isotopes:

During our discussion we have neglected mention of the use of stable isotopes. This is neither because they are not useful as tracers nor because they are not available. Rather, they do not have as versatile qualifications for tracer utilization as radioisotopes and to date they have not received as widespread attention. Stable isotope detection requires thousands to many millions of times more atoms than radioisotope detection. It follows then that the stable isotope technique does not permit as large experimental dilutions as does the radioisotope technique. Also the detection instrument for stable isotopes, the mass spectrometer, is a large and expensive piece of equipment which until recently has not readily been available on the open market. This then accounts for the somewhat limited utilization of stable isotopes.

Since stable isotopes have no radioactivity, they can be distinguished only by small differences in their weight. The use of a stable isotope as a tracer, therefore, is dependent upon a determination of its weight in the presence of other stable isotopes of the same element. In order to make such a measurement, the tracer isotope must be present in a concentration greater than its normal abundance. The stable isotopes of carbon 12 and carbon 13, for instance, are found in all naturally occurring carbon in the respective abundances of 98.9 per cent and 1.1 per cent. If carbon 13 is concentrated to several times this abundance, it is possible to use it as a tracer. On the other hand carbon 12 cannot be sufficiently concentrated above its normal abundance for tracer utilization. Generally, an isotope has to be concentrated several times its normal abundance to be useful as a tracer. As an example chlorine has two stable isotopes, but the one of least abundance, chlorine 37, has a natural abundance of 24.6 per cent which precludes its being concentrated for great usefulness as a tracer.

Deuterium (heavy hydrogen), boron 10, and oxygen 18 are being distributed through Atomic Energy Commission facilities and used in many applications. Carbon 13 and nitrogen 15 have for many years been available from commercial sources and used in tracer studies with much success. Only recently, however, has it been possible to obtain electromagnetically concentreted stable isotopes of a wide variety of elements (**76**). Isotopes of this group, comprising elements from lithium to lead, are now being concentrated in the same type of large scale mass spectrometers which were used during the war to concentrate uranium 235. These heretofore unavailable stable isotopes are now procurable in limited quantities through the Commission.

Stable isotopes are quite useful for tracer studies in many fields. In some instances they are used instead of radioisotopes where the presence of radiation would not be permissable—in other cases where no suitable radioactive isotope is available. An example of the latter case is oxygen, for which the only useful tracer is the stable oxygen 18. This isotope will be very useful in studying and analyzing certain oxygen-containing organic compounds, as well as in many biological investigations.

Although stable isotopes can broaden the over-all scope of the isotope technique, their widespread utilization is faced with three limitations: (*1*) because their identity is based on their concentration or enrichment, their value as tracers will be limited by the dilution caused by the experiment, (*2*) in some cases only one stable isotope of an element exists, and (*3*) their concentration and presence must be measured with a mass spectrometer, an instrument now commercially available but still expensive. Nevertheless the use of stable isotopes in tracer research and in process control will no doubt become increasingly extensive and important.

This review of isotope utilizations has not been complete—in fact it has only touched on a few applications in only one of their many fields of usefulness, namely in industry. It is hoped, however, that the summary has been sufficiently complete to give an understandable over-all picture of the ways in which isotopes may be used.

GENERAL CONCLUSIONS

I should like to leave a few general thoughts with you, not only on isotopes but on atomic science and atomic energy.

The rapidly expanding world population and increasing standard of living is continually placing greater demands on industry. Possibly the foremost of these demands is for continued and improved application of raw materials and resources. It will call for the most efficient tapping of natural resources, the most economical processing of materials, and the greatest utilization of all the final products. It is hoped that the opportunity for research and application with isotopes will be utilized fully in tackling the all-important problems in your field of industry—testing materials—and indeed the whole of industry.

In my introductory remarks I pointed out that advances in production and material things have been preceded by advances in fundamental science—that the two go ahead together hand in hand. I should like to close with further remarks on this theme in relation to atomic energy.

It is natural that great emphasis has been placed on atomic energy as a powerful and deadly instrument of warfare, and on atomic energy as a vast potential source of useful power. These aspects are tremendously important and must be fully understood. It is equally important, however, that the lay public—and technical persons as well—not lose sight of the fact that atomic energy is much more than power, either constructive or destructive. It must also be viewed in the light of its broader aspects, as a vigorous new scientific and technical development from which will pour forth new knowledge of all things physical. Atoms enter into all material things and processes—into the structure and functioning of machines, plants, animals and man. Study of the atom, its forces, its structure and its behavior, down to the details of subatomic or nuclear phenomena, is basic to all fields of science and development.

Industry has not only been giving strong support to fundamental research in all fields, but "long range" and fundamental investigations have become an increasing part of its own research programs. Considerable industrial participation in the basic development of atomic energy has already taken place, and a good share of the future progress in all phases of atomic energy will depend on the degree of increased interest and participation shown by industry in "pure" research in this field (**77**).

Industry of course has the further, very important rôle of using the results of fundamental research to develop applications of benefit to human living—not only benefits to material comfort but also to over-all human welfare. This rôle is an especially stimulating challenge in the case of atomic energy.

Atomic energy in the form of its by-products, radioisotopes, offers to industry a tool to aid in carrying out these two important rôles. On the one hand they can aid the industrial research worker in studying the basic principles underlying the chemical and physical reactions of his processes. On the other they can assist the engineer in development and production to meet more rigid specifications, and therefore to manufacture a better product.

Neither the public nor industrial management should be misled into thinking that all research should appear to have some definite benefits or applications, some immediate prospects of use—

or misled into thinking that routine programmatic research is truly fundamental research, the kind of research that may lead to complete new concepts or developments. There must be more general understanding of what fundamental science is and how its research is carried out. In particular, the principles of true scientific thought and the attitudes of the scientist must be understood. Public and industrial administrators must realize the heavy responsibility they have in these less evident respects—in promoting the health and vigor of our fundamental research in all fields, and especially in atomic energy.

Perhaps one of the most important things to recognize in supporting fundamental research is that we do not support just a program and a laboratory. Rather, research is really the support of men capable of new ideas—men expecially chosen for their abilities in reasoning and imagination, for their enthusiasm for the scientific method, and for their confidence in attaining results.

Results of fundamental research can no more be predicted than the new ideas that will arise in men through stimulation of their search for knowledge. Who could have predicted that Roentgen's and Becquerel's independent studies of fluorescence would have led to the great discoveries of the X-ray, radioactivity, and the electron—and then that these would have led to all their innumerable applications in medicine, technology, education and industry? Who could have predicted that only two decades of studying the nucleus of the atom would have led to such world-wide consequences?

Who can predict where studies of the newly found subatomic particles—the mesons—will lead? Who can predict what will come from studies of photosynthesis—the little known process by which plants capture the great available energy of sunlight and store it for future use?

Your field, of course, is in materials—in testing materials to make better products and to make more efficient use of our natural resources. Atomic energy is a new and revolutionary source from which will flow an ever increasing number of materials and processes. If we can develop and direct the products from this newly found resource toward constructive goals, then as truly as the abundant harvests and great resources of our country have flowed out to enrich the whole world, so will flow the fruitful harvests of atomic science and the vast resources of atomic power.

Finally, I wish to close with these thoughts on our rôle in the "Atomic Age." As citizens—as human beings—whether primarily involved in industry, agriculture, education, medicine, or other fields of endeavor, we must not lose sight of atomic energy's great challenge to humanity—the challenge to prevent war and unbelievable destruction and to promote the use of this new force for human welfare. This challenge is not just for statesmen or scientists, but for each one of us. It is not something too complicated or too secret, or too fearful, or too hopeless, for the average person to "tackle."

You, who have special scientific and technical knowledge, can of course do much to study the problems and facts, on both the constructive and destructive aspects, and keep yourself and your friends well informed. Next, through our democratic procedures and representation you can urge wide and vigorous support of the measures developed by our well tested, common sense methods of unbiased reasoning. Finally, you can help to master the fears in yourselves and others, which if allowed to go uncontrolled may shake our faiths—our faiths in each other, in our way of life, in our free-enterprise system, in our form of government, in our spiritual and moral beliefs—for it is in these faiths, as much or more than in our mighty weapons and technology, that we are truly strong and capable of leading the world to peace and greater human welfare.

REFERENCES

(1) *Westinghouse Engineer*, December, 1945.

(2) A. V. Grosse and G. H. Cady, "Properties of Fluorocarbons," *Industrial and Engineering Chemistry*, Vol. 39, p. 367 (1947).

(3) "Fluorocarbons," *Chemical Engineering*, Vol. 55, p. 78 (1948).

(4) F. Landau and R. Rosen, "Industrial Handling of Fluorine," *Industrial and Engineering Chemistry*, Vol. 39, p. 281 (1947).

(5) "Fluorine Chemistry Achieves Commercial Stature," *Chemical Industries*, Vol. 59, p. 1006 (1946).

(6) S. G. Turnbull, A. F. Benning, G. W. Reldmann, A. L. Linch, R. C. McHarness, and M. K. Richards, "Analysis and Disposal of Fluorine," *Industrial and Engineering Chemistry*, Vol. 39, p. 286 (1947).

(7) S. C. Ogburn, "Fluorine, An Important Material for Postwar Development," *Journal of Chemical Education*, Vol. 24, p. 314 (1947).

(8) C. D. Goodman, "Science and Engineering of Nuclear Power," Addison Wesley Press, Cambridge, Mass. (1947).

(9) A. W. Kramer, "The Significance of the Nuclear Reactor in Atomic Development," *Power Generation*, Vol. 90, March, 1948.

(10) J. R. Menke, "Nuclear Fission As Source of Power," *MDDC 1104* (MDDC reports, recently changed to AECD, represent unclassified and declassified reports prepared and issued by the Manhattan District and the Atomic Energy Commission.)

(11) Isotopes Division, *Catalog and Price List No. 2*, September, 1947, United States Atomic Energy Commission, P. O. Box E, Oak Ridge, Tenn.

(12) P. C. Aebersold, "Recent Developments in the Availability of Isotopes," Symposium on Isotopes in Biology and Medicine, The University of Wisconsin Press, Madison, Wis. (1948).

(13) United States Atomic Energy Commission, *Fourth Semiannual Report*, 1948 (For sale by the Superintendent of Documents, United States Government Printing Office, Washington 25, D. C.)

(14) P. C. Aebersold, "The Cyclotron: A Nuclear Transformer," *Radiology*, Vol. 39, p. 513 (1942).

(15) C. A. Thomas, "Radioisotopes: New Tools for Science," *Chemical and Enigneering News*, Vol. 25, p. 1572 (1947).

(16) "Business in Isotopes," *Fortune*, Vol. 36, December, 1947, p. 121.

(17) "The Isotope Distribution Program," *Science*, Vol. 106, August, 1947, p. 175.

(18) H. S. Taylor, "Isotopes in Chemistry and Metallurgy," *Metal Progress*, Vol. 49, p. 1207 (1946).

(19) F. A. Vick, "Recent Advances in Science," *Science Progress*, No. 139, July, 1947.

(20) L. I. Katzin, "Industrial Uses of Atomic Energy," *Scientific American* Vol. 98, February, 1946.

(21) P. C. Aebersold, "Isotopes for Industry," *Federal Science Progress, No. 1*, p. 3, May, 1947.

(22) A. R. Gardner, "Putting Atoms to Work in Your Plant," *Modern Industry*, Vol.50 February 15, 1948.

(23) A. R. Gardner, "How to Buy and Use Isotopes," *Modern Industry*, Vol. 40, March 15, 1948.

(24) J. M. Cork, "Radioactivity and Nuclear Physics," Van Nostrand Inc., New York, N. Y. (1947).

(25) E. Pollard and W. L. Davidson, Jr. "Applied Nuclear Physics," John Wiley and Sons, Inc., New York, N. Y. (1942).

(26) H. P. Knauss, "Slide Rule Calculations of Radioactivity Decay," *Science*, Vol. 107, p. 324 (1948).

(27) R. D. Evans, "Radioactivity Units and Standards," *Nucleonics*, Vol. 1, No. 2, p. 32 (1947).

(28) R. E. Lapp and H. L. Andrews, "Nuclear Radiation Physics," Prentice-Hall, Inc., New York, N. Y. (1948).

(29) M. D. Kamen, "Radioactive Tracers in Biology," Academic Press, Inc., New York, N. Y. (1947).

(30) P. W. McDaniel, "The Measurement of Radiation," *Proceedings*, Auburn Conference on the Use of Radioactive Isotopes

in Agricultural Research, Edward Brothers, Inc., Ann Arbor, Mich. (1948).

(31) C. D. Coryell, "Fundamental Principles of the Detection and Measurement of Radioactivity," Symposium on Isotopes in Biology and Medicine, The University of Wisconsin Press, Madison, Wis. (1948).

(32) W. F. Libby, "Measurement of Radioactive Tracers," *Industrial and Engineering Chemistry Analytical Edition*, Vol. 19, p. 2 (1947).

(33) C. R. Schwob, "Hot Laboratory Instrumentation," *Instruments*, Vol. 20, p. 712 (1947).

(34) L. R. Keller, "Gamma Ray Detection of Internal Corrosion," *Petroleum Engineering*, Vol. 18, p. 211 (1947).

(35) L. Brown, "Economics of Industrial Radiography," *Western Metals*, Vol. 4, No. 9, p. 21 (1946).

(36) R. Halmshaw, "Technique of Gamma Radiography," *Engineering*, Vol. 162, No. 4206, p. 169 (1946).

(37) P. S. Koltunor, "Radioscopy of Industrial Products by Gamma Rays," *Vestnik Inzheneror i Technikor*, No. 6, June, 1947.

(38) J. C. Chaston, "Gamma-Ray Radiography for Inspection Work; A Comparison with X-Ray Technique," *Aircraft Production*, Vol. 9, p. 309 (1947).

(39) J. E. Wirth, "Essential Safe-Guards in Production and Use of Atomic Energy," *Occupational Medicine*, Vol. 2, p. 428 (1946).

(40) K. Z. Morgan, "Radiation Safety: A New Industrial Problem," *Scientific American*, Vol. 178, p. 5 (1948).

(41) "General Rules and Procedures Concerning Radioactive Hazards," *Nucleonics*, Vol. 1, No. 4, p. 60 (1947).

(42) G. W. Morgan, "Protective Precautions in Handling of Radioactive Materials," *Proceedings*, Auburn Conference on the Use of Radioactive Isotopes in Agricultural Research, Edward Brothers, Inc., Ann Arbor, Mich. (1948).

(43) W. H. Sullivan, "Control of Radioactivity Hazards," *Chemical and Engineering News*, Vol. 25, p. 1862 (1947).

(44) P. C. Tompkins, "Laboratory Handling of Radioactive Materials," *MDDC 1414*.

(45) H. A. Levy, "Aspects of the Design of Radiochemical Laboratories," *Chemical and Engineering News*, Vol. 24, p. 3168 (1946).

(46) F. C. Henriques, Jr., and A. P. Schreiber, "Administration and Operation of a Radiochemical Laboratory," *Nucleonics*, Vol. 2, No. 3 (1948).

(47) K. Block and H. S. Anker, "An Extension of the Isotope Dilution Method," *Science*, Vol. 107, p. 228 (1948).

(48) F. C. Henriques, Jr. and C. Margnetti, "Radioactive Isotope Dilution Procedures," *Industrial and Engineering Chemistry*, Vol. 18, p. 476 (1946).

(49) J. T. Kummer, "Production of Radioactive Carbon Monoxide from Barium Carbonate," *Journal of the American Chemical Society*, Vol. 69, p. 2239 (1947).

(50) J. T. Kummer, L. C. Browning and P. H. Emmett, "Thermodynamic Calculations Concerning the Possible Participation of the Carbides of Iron as Intermediate in the Fischer-Tropsch Synthesis," *The Journal of Chemical Physics*, Vol. 16, p. 739 (1948).

(51) G. Hardy, "Metalworking Industry Gets Real Benefit from Atomic Pile," *Iron Age*, Vol. 160, p. 108 (1947).

(52) J. J. Harwood, "Tracers in Metallurgy," *Nucleonics*, Vol. 2, No. 1, p. 57 (1948).

(53) "Source of Sulfur Entering Steel Disclosed by Radioactive Tracers," *Steel*, Vol. 122 (1948).

(54) S. E. Eaton, "A Study of the Role of Sulfur in the Coking Process for the Steel Industry," *Industrial Bulletin* (A. D. Little, Inc.) April, 1948.

(55) J. K. Stanley, "Some Possibilities of Radioactive Tracers in Metallurgy," *Nucleonics*, Vol. 1, No. 2, p. 70 (1947).

(56) J. K. Stanley, "A Carborizing Experiment with Radioactive Carbon 14," *Metal Progress*, Vol. 52, p. 227 (1947).

(57) E. S. Kopecki, "Radioactive Tracers in Metallurgical Research," *Iron Age*, Vol. 160 (1947).

(58) H. C. Plant, "Metallurgy and Atomic Energy," *Metallurgia*, Vol. 37, pp. 15, 137, 178, 248 (1947, 1948).

(59) R. F. Bacher, "The Metallurgist and Atomic Energy," *Metal Progress*, Vol. 52, p. 800 (1947).

(60) G. C. Kuczynski, "A New Method of Measuring Diffusion Coefficient in Solids with Radioactive Tracers," *Journal of Applied Physics*, Vol. 19, p. 308 (1948).

(61) W. E. Jones, "Memorandum on the Use of Radioisotopes as Tracers in High Temperature Alloys," *Bulletin of the General Electric Company*, March 17, 1947.

(62) W. E. Jones, "Use of Radioactive Tracer Elements to Identify Minor Constituents in High Temperature Materials," *Bulletin* *of the General Electric Company*, September 10, 1948.

(63) J. T. Burwell, "Radioactive Tracers in Friction Studies," *Nucleonics*, Vol. 1, No. 4, p. 38 (1947).

(64) B. W. Sakman, J. T. Burwell, Jr., and J. W. Irvine, "Measurements of the Adhesion Component in Friction by Means of Radioactive Indicators," *Journal of Applied Physics*, Vol. 15, p. 459 (1944).

(65) A. W. Knoerr, "Geiger-Mueller Counters Applied to Mining," *Engineering and Mining Journal*, Vol. 148, No. 7, p. 92 (1947).

(66) V. J. Mercier, "Radioactivity Well Logs, Interpretation and Application," *Oil Weekly*, Vol. 123, No. 7, October 14, 1946, p. 56; Vol. 123, No. 8, October 21, 1946, p. 41.

(67) R. B. Rose, "Radioactive Exploration," *Mining World*, Vol. 8, No. 9, August, 1946, p. 46.

(68) H. G. Doll and H. F. Schwede, "Radioactive Markers in Oil Field Practice," *Oil Weekly*, Vol. 50, March 17, 1947.

(69) J. D. Haseman, "Significance of Atomic Energy to Petroleum Problems," *Petroleum Engineer*, No. 18, April, 1947, p. 70.

(70) R. E. Fearon, "Influence of Atomic Science on Petroleum," *Oil Weekly*, Vol. 123, No. 7, October 14, 1946, p. 47.

(71) "Radioactive Tracers: How They Can Be Used In Flotation Research," *Engineering and Mining Journal*, Vol. 149, p. 53 (1948).

(72) R. H. Ramsey, "Research and Development Open New Phase in Milling," *Engineering and Mining Journal*, Vol. 149, p. 104 (1948).

(73) A. P. Schreiber, "Chemical Process Control with Radioactivity," *Nucleonics*, Vol. 2, No. 1, p. 33 (1948).

(74) O. J. M. Smith, "Beta-ray Thickness Gage for Sheet Steel," *Electronics*, Vol. 20, p. 106 (1947).

(75) A. H. W. Aten, Jr. and F. A. Heyn, "The Use of Isotopes as Tracers," *Phillips Technical Review*, Vol. 8, p. 296 (1946).

(76) "More Stable Isotopes are Available," *Chemical Engineering*, Vol. 55, p. 186 (1948).

(77) "Industry Cooperates in Atomic Research," *Chemical and Engineering News*, Vol. 26, p. 254 (1948).

HIGH-TEMPERATURE PROPERTIES OF ROTOR DISKS FOR GAS TURBINES AS AFFECTED BY VARIABLES IN PROCESSING[*]

By J. W. Freeman,[1] Howard C. Cross,[2] E. E. Reynolds,[1] and Ward F. Simmons[2]

Synopsis

This paper shows the results of high-temperature tests on 24 large forged disks of 8 heat-resisting alloys. The materials studied ranged from relatively low-alloyed materials such as Timken 16-25-6, CSA, EME, and 19-9DL alloys through the more highly-alloyed materials low-carbon N-155, S-590, S-816, and Inconel-X alloys. Short-time tension, rupture, creep, and stress-time for total deformation characteristics were determined at 1200, 1350, and 1500 F.

At the start of the work, the testing was begun to obtain the numerical high-temperature strengths of disks of only a few compositions and to establish the degree to which the properties of experimental bar stock could be reproduced in the large forgings required for rotor disks. Now a large number of disks of various compositions have been tested, and the indicated principles of the effects of processing variables and heat treatment appear to be of relatively greater importance than the numerical strengths.

Properly hot-cold-worked or as-forged materials, except for Inconel-X, have the best properties up to 1200 F. At 1350 F., solution treated and aged materials have properties equal to or better than the hot-cold-worked and forged materials, and at 1500 F. are definitely superior.

The requirements for brevity in a synopsis result in generalizations which cannot help but fail to indicate many modifying circumstances. To those more than casually interested, a careful inspection of the entire paper is respectfully suggested.

In the development of new alloys for service in the high-temperature components of gas turbines, experimental investigations have been necessary to establish the degree to which the propties of experimental bar stock can be reproduced in the large forgings required for rotor disks. Information of this type is presented for eight alloys, Timken 16-25-6, CSA, 19-9DL, EME, low-carbon N-155, S-590, S-816, and Inconel-X in the form of forged disks. For most of the materials, several disks were tested as representative of various procedures for producing such disks. While most of the forgings are fairly large in size, they are primarily representative of the disks which are used in gas turbines for aircraft.

Because the rims of the rotor disks of the type investigated operate at the highest temperature, the testing program was largely confined to specimens cut from the rims in a radial direction.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Research Engineer and Research Associate, respectively, Engineering Research Institute, University of Michigan, Ann Arbor, Mich.

[2] Supervisor and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

All of the disks were tested at room temperature and 1200 F., and in addition the testing temperatures were extended to 1350 and 1500 F. for the more highly alloyed materials. The data have been summarized to show the short-time tensile properties, rupture-test characteristics, creep strengths and stress-time for total deformation properties.

The investigations on which this paper is based were done under contract for the National Advisory Committee for Aeronautics, for the National Defense Research Committee, and for the Office of Naval Research, United States Navy. The paper constitutes a summary of the 12 individual reports (**1–12**)[3] which have been submitted to the sponsor organizations. These individual reports have been given limited circulation by these organizations.

TABLE I.—CHEMICAL COMPOSITION OF DISKS TESTED.

Alloy	Disk Number	Chemical Composition, per cent													
		C	Mn	P	S	Si	Cr	Ni	Co	Mo	W	Cb	N	Ti	Other
Timken 16-25-6......	1	0.10	1.24	0.022	0.022	0.72	16.56	25.75	...	5.80	...	...	0.12	...	
	2	0.094	1.90	0.024	0.012	0.70	16.60	25.08	...	5.86	...	...	0.141	...	
	3	0.096	1.74	0.019	0.018	0.62	17.14	25.62	...	6.18	...	...	0.182	...	
	4	0.11	1.49	0.023	0.023	0.64	15.67	26.25	...	6.25	...	...	0.111	...	
	5	0.12	1.12	0.017	0.014	0.71	15.90	26.20	...	6.30	...	...	0.110	...	
CSA................	6	0.25	4.14	0.017	0.023	0.25	18.32	5.76	...	1.46	1.51	0.95	...	...	
	7, 8	0.42	4.47	0.030	0.016	0.41	18.06	5.20	...	1.30	1.20	0.28	...	...	
EME................	9	0.12	0.48	0.015	0.028	0.73	19.72	11.60	...	...	3.38	1.30	0.127	...	
19-9DL.............	10, 13, 14	0.33	1.14	0.016	0.015	0.65	19.10	9.05	...	1.35	1.14	0.35	...	0.16	
	11, 12	0.29	1.12	0.018	0.010	0.69	19.23	9.03	...	1.27	1.58	0.38	...	0.43	
Low-Carbon N-155..	15	0.15	1.74	...	...	0.37	21.66	19.40	19.02	2.76	1.90	0.79	0.14	...	
	16, 17, 18	0.07	1.68	...	...	0.60	20.80	20.60	20.07	2.94	2.67	1.05	0.125	...	
S-590..............	19, 20, 21	0.45	1.44	0.015	0.018	0.56	19.76	19.05	20.20	4.03	4.08	3.35	...	...	
S-816..............	22, 23	0.38	0.50	...	...	0.53	19.80	20.57	42.71	3.90	4.76	3.95	...	...	Fe 2.87
Inconel-X...........	24	0.05	0.51	...	0.007	0.39	14.61	73.42	...	...	...	1.04	...	2.33	Fe 6.90, Al 0.67, Cu 0.04

Previous reports on testing of bar stock of some of these same alloys have already been presented to the Society (**13, 14**).

The investigation of these disks for the N.A.C.A. was conducted at the Engineering Research Institute of the University of Michigan and for the U. S. Navy and the N.D.R.C. at Battelle Memorial Institute.

TEST MATERIALS

The chemical compositions of the disks are listed in Table I. The Timken 16-25-6 alloy is the well-known alloy containing 16 per cent chromium-25 per cent nickel-6 per cent molybdenum which was used for production of turbosupercharger rotor disks and is now being used for rotor disks in some of the jet aircraft engines. This alloy is customarily used in the hot-cold-worked condition with the final reduction applied in the temperature range 1200 to 1350 F.

The CSA alloy is a modified 18 chromium-8 nickel steel in which half of the nickel has been replaced by manganese, and molybdenum, tungsten, and columbium have been added. It was of interest because of its possibility as a replacement for more highly alloyed materials.

The EME alloy is a modified 18 chromium-8 nickel steel, strengthened by additions of tungsten, columbium, and nitrogen. One of the claims made for this alloy is that it lends itself to the production of high-quality forgings.

---

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 587.

The 19-9DL alloy is also a modified 18 chromium-8 nickel steel, with somewhat higher carbon content (0.28 to 0.35 per cent) and strengthened by moderate additions of molybdenum, tungsten, columbium, and titanium. This alloy is also being used for gas turbine rotor disks as hot-cold worked directly after forging or following a solution treatment.

Low-carbon N-155, which is a more highly alloyed material than those mentioned previously, contains about 20 per cent cobalt in addition to the 20 per cent of chromium and nickel and is strengthened by additions of 3 per cent molybdenum, 2 per cent tungsten, and 1 per cent columbium.

The S-590 alloy is quite similar to low-carbon N-155 alloy, and differs in that it contains higher carbon and larger additions, approximately 4 per cent each, of molybdenum, tungsten, and columbium.

The S-816 alloy is the most highly alloyed material tested and differs from S-590 alloy in that cobalt replaces the iron content, making this a cobalt-base rather than an iron-base alloy.

The Inconel-X alloy is a low-carbon nickel-base Ni-Cr-Fe alloy of the precipitation-hardening type in which strengthening results from additions of titanium, aluminum, and columbium when subjected to suitable solution and aging treatments.

The more highly alloyed materials low-carbon N-155, S-590, S-816, and Inconel-X were included to provide information on materials thought most likely to show usable properties in the higher temperature range of 1350 to 1500 F. These materials show their best long-time properties at these temperatures in the solution-treated and aged condition.

Because it has been recognized for some time that the properties of alloys of the type considered in this investigation are influenced by processing conditions and heat treatment, this type of information is given in Table II as completely as it could be obtained from the manufacturers. In several cases they were among the first disks forged from new alloys and as such should be considered only as representative of experimental development and not the ultimate properties which may have been or could be subsequently obtained with more experience in selecting forging and heat treatment conditions for large forgings.

The disks tested represent various combinations of treatments. Some variation in size is represented. Some of the disks were contour-forged to nearly the finish dimensions. Others were simply flat "cheese" type disks. A few disks were tested in the as-forged condition. Hot-cold work to improve properties was used in some cases both on as-forged materials and subsequent to solution treatments. Other disks were aged, or solution-treated and aged, subsequent to hot-forging because prior experience with the alloys indicated that they develop their best properties with such treatments.

The treatments given the disks were based on both the best judgment of the producers for developing the properties desirable in disks for gas turbines, and on indicated experimental treatments. The latter treatments were used primarily to ascertain what properties could be developed in large disks with such treatments. For instance, as-forged disks of S-590 and S-816 alloys were included for experimental purposes

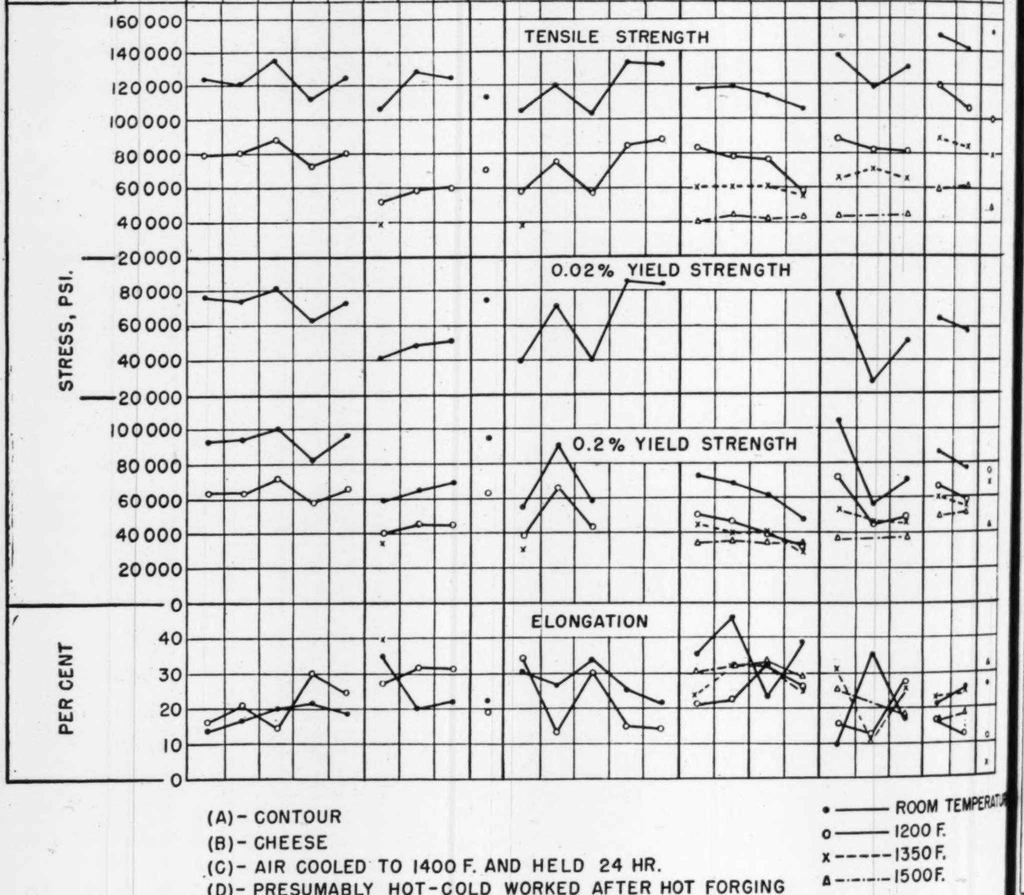
TABLE II.—SOURCE, FABRICATION, AND HEAT TREATMENT OF DISKS.

Alloy	Disk	Alloy Manufacturer	Disk Forged by	Type of Heat	Fabrication Procedure	Approximate Finished Size of Disk		Heat Treatment	
						Diameter, in.	Thickness, in.	Solution Heat Treatment	Final Heat Treatment
Timken 16-25-6	No. 1......	The Midvale Co.	The Midvale Co.		Contour forging, presumably hot-cold worked after hot-forging	20	3½	None	
	No. 2......	Timken Roller Bearing Co.	Canton Drop Forging & Mfg. Co.	Electric arc heat	Contour forging, hot-forged in 3 heats from 2000 F., air cooled. Hot-cold worked at 1250 F.	20	3½	None	1200 F.—10 hr.
	No. 3......	Timken Roller Bearing Co.	Canton Drop Forging & Mfg. Co.		Contour forging, hot-forged from 2000 F. Solution treated prior to hot-cold working at 1250 F.	20	3½	2150 F., 2 hr., water quenched[a]	1200 F.—10 hr.
	No. 4......	The Midvale Co.	The Midvale Co.		Cheese forging, presumably hot-cold worked after hot-forging	28	4		
	No. 5......	The Midvale Co.	The Midvale Co.	2620-lb. ingot, 16-in. diameter bottom poured	Contour forging, hot-forged from 2000 F., hot-cold worked at 1275 F.	28	2⅜		1200 F
CSA	No. 6......	Crucible Steel Co.	Crucible Steel Co.	Induction heat, 15-in. ingot	Cheese forging, ingot hot-forged from 2050 F. to 9-in. square billet, upset to disk in six heats from 2100 F. with final final finishing at 1400 F.	20⅛	3 5/16	None	1200 F.—4 hr.
	No. 7......	Crucible Steel Co.	Crucible Steel Co.	Induction heat, 15-in. square ingot	Cheese forging, ingot hot-forged to 9-in. square billet, upset to disk in one heat from 2150 F. with final finishing at 1650 F. Solution treated prior to hot-cold working at 1420 to 1250 F., reduced from 3⅞ to 3½-in. thick (10%)	20	3½	2050-2100 F., air cooled[a]	1200 F.—4 hr.
	No. 8......	Crucible Steel Co.	Crucible Steel Co.	Induction heat, 15-in. square ingot	Same as disk No. 7	20	3½	2050-2100 F., air cooled to 1400 F. and aged 24 hr. Air cooled to room temp.[a]	1200 F.—4 hr
EME	No. 9......	The Midvale Co.	The Midvale Co.	Arc-type basic electric, 2600 lb. 17-in. diameter ingot	Contour forging, hot-forged to 10¼-in. diameter billet, hot-forged from 2050-2100 to 1700 F., hot-cold worked at 1240 F.	21	4½	None	1200 F.—4 hr.

19-9DL	No. 10	Universal-Cyclops Steel Corp.	Universal-Cyclops Steel Corp.	Arc-furnace 10,000-lb. heat	Cheese forging, directly upset from billet, finishing temp. 1640 F.	19¾	3¼	None	1200 F.
	No. 11	Universal-Cyclops Steel Corp.	Canton Drop Forging and Mfg. Co.	Basic electric arc, 24,000-lb. heat, 15½-in. square, 2600-lb. ingot	Contour forging, hot-forged in 3 heats from 2100 F. Solution treated prior to hot-cold working at 1250 F.	20	3½	2150 F., 2 hr., quenched in warm water[a]	1200 F.—10 hr.
	No. 12	Universal-Cyclops Steel Corp.	Canton Drop Forging and Mfg. Co.	Basic electric arc, 24,000-lb. heat, 15½-in. square, 2600-lb. ingot	Same as disk No. 11, except for cold working at 1650 F.	20	3½	2150 F., 2 hr., quenched in warm water[a]	1200 F.—10 hr.
	No. 13	Universal-Cyclops Steel Corp.	The Steel Improvement and Forge Co.	Arc furnace 10,000-lb. heat	Contour forged disk hot-forged from billet hot-cold worked at 1300–1350 F. in closed contour dies	12	1	None	1200 F.—4 hr.
	No. 14	Universal-Cyclops Steel Corp.	The Steel Improvement and Forge Co.	Arc furnace 10,000-lb. heat	Same as disk No. 13 except solution treated prior to hot-cold work	12	1	2100 F., 1 hr., air cooled[a]	1200 F.—4 hr.
Low-Carbon	No. 15	Universal-Cyclops Steel Corp.	Universal-Cyclops Steel Corp.	Arc furnace 10,000-lb. heat	Cheese forging, 9-in. billet directly upset with finishing temperature of 1630 F.	21	3¼	None	1200 F.—2 hr.
	No. 16	Crucible Steel Co.	Crucible Steel Co.	Induction furnace, 2000-lb. heat	Cheese forging, 9-in. billet upset with finishing temperature of 1500 F.	14¾	4⅜	None	None
	No. 17	Crucible Steel Co.	Crucible Steel Co.	Induction furnace, 2000-lb. heat	Same as disk No. 16, then solution treated and aged	14¾	4⅜	2200 F.—water quenched	1500 F.—24 hr.
	No. 18	Crucible Steel Co.	Crucible Steel Co.	Induction furnace, 2000-lb. heat	Same as disk No. 16, then solution treated and aged	14¾	4⅜	2200 F.—water quenched	1350 F.—24 hr.
S-590	No. 19	Allegheny-Ludlum Steel Corp.	Allegheny-Ludlum Steel Corp.	Two-ton electric-arc furnace, 12-in. ingot	Cheese forging, hot-forged from 2250 F. to 9-in. square billet, upset from 2250 F. to rough 4-in. disk, hot-forged from 2250 to 3¾-in. thick and from 2000 F. to 3⅜-in. thick	18	3⅜	None	1400 F.—16 hr.
	No. 20	Allegheny-Ludlum Steel Corp.	Allegheny-Ludlum Steel Corp.	Two-ton electric-arc furnace, 12-in. ingot	Cheese forging, hot-forged from 2250 F. to 9-in. square billet, upset from 2250 F. to 3-in. thick	18	3	2300 F., 3¼ hr., water quenched	None
	No. 21	Allegheny-Ludlum Steel Corp.	Allegheny-Ludlum Steel Corp.	Two-ton electric-arc furnace, 12-in. ingot	Same as disk No. 20	18	3	2300 F., 3¼ hr., water quenched	1400 F.—16 hr.
S-816	No. 22	Allegheny-Ludlum Steel Corp.	Allegheny-Ludlum Steel Corp.	Electric-arc 5000-lb. heat 12-in. square ingot	Cheese forging, hot-forged from 2300 F. to 9½-in. square billet, upset from 2250 F. to 3$\frac{5}{16}$-in. thick	17	3$\frac{5}{16}$	None	1400 F.—16 hr.
	No. 23	Allegheny-Ludlum Steel Corp.	Allegheny-Ludlum Steel Corp.	Electric-arc 5000-lb. heat 12-in. square ingot	Same as disk No. 22	17	3$\frac{5}{16}$	2300 F., 2½ hr., water quenched	1400 F.—16 hr.
Inconel-X	No. 24	International Nickel Co., Inc.	General Electric Co.	Induction furnace 4800-lb. heat 18-in. square ingot	Cheese forging, hot-forged from 2225 F. to 9½-in. diameter billet, turned to 8½-in. diameter, upset in 5 heats from 2225 to 1800 F.	22	3½	2100 F., 4 hr., water quenched	1550 F.—24 hr., then 1300 F.—20 hr.

[a] Treatment prior to hot-cold work.

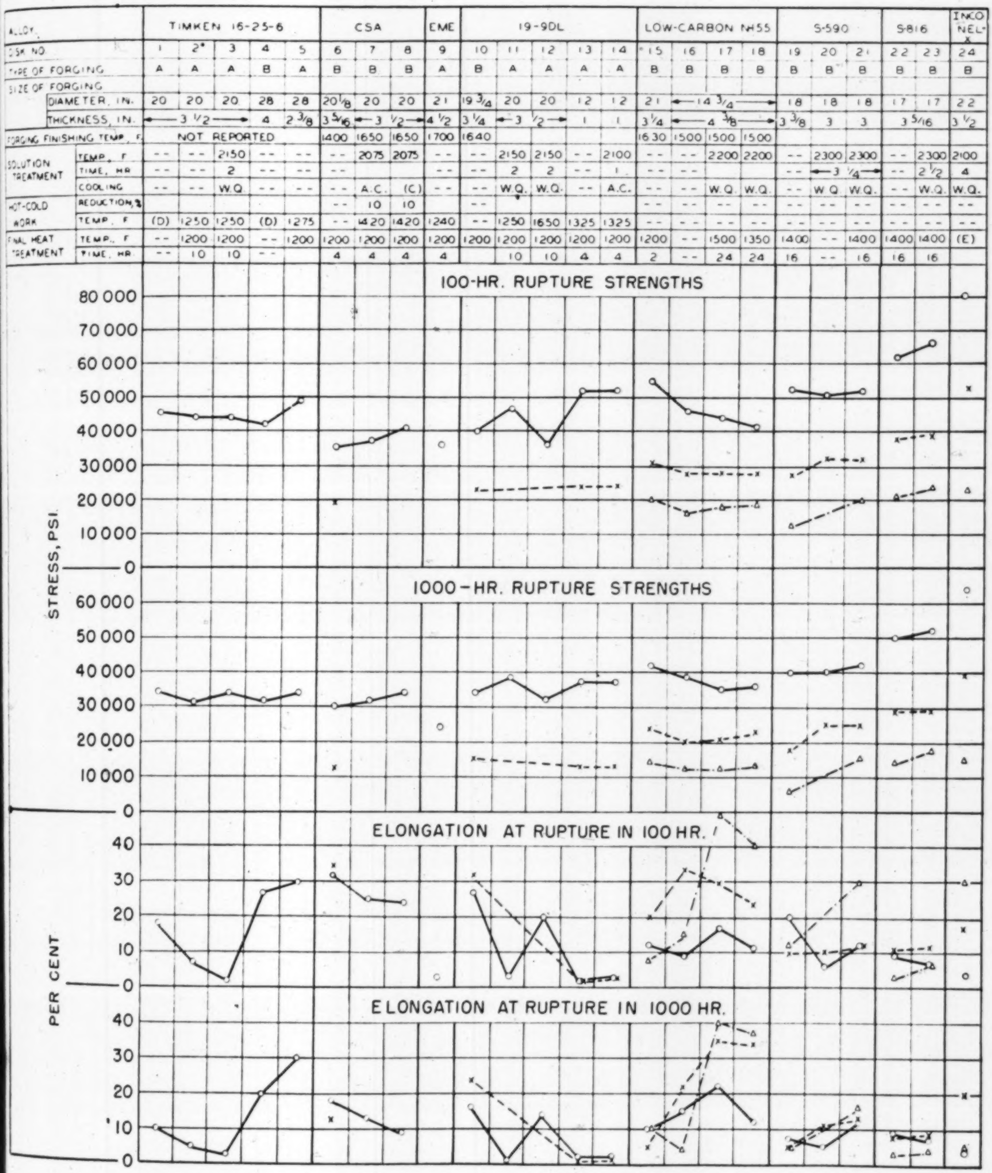
ALLOY		TIMKEN 16-25-6					CSA			EME	19-9DL					LOW-CARBON N155				S-590			S-816		INCONEL X
DISK NO.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
TYPE OF FORGING		A	A	A	B	A	B	B	B	A	B	A	A	A	A	B	B	B	B	B	B	B	B	B	B
SIZE OF FORGING																									
	DIAMETER, IN.	20	20	20	28	28	20 1/8	20	20	21	19 3/4	20	20	12	12	21	← 14 3/4 →			18	18	18	17	17	22
	THICKNESS, IN.	← 3 1/2 →			4	2 3/8	3 5/16	← 3 1/2 →		4 1/2	3 1/4	← 3 1/2 →		1	1	3 1/4	← 4 3/8 →			3 3/8	3	3	3 5/16		3 1/2
FORGING FINISHING TEMP., F		NOT REPORTED					1400	1650	1650	1700	1640					1630	1500	1500	1500						
SOLUTION TREATMENT	TEMP., F	--	--	2150	--	--	--	2075	2075	--	--	2150	2150	--	2100	--	--	2200	2200	--	2300	2300	--	2300	2100
	TIME, HR.	--	--	2	--	--	--			--	--	2	2	--	1	--	--			--	← 3 1/4 →		--	2 1/2	4
	COOLING	--	--	W.Q.	--	--	--	A.C.	(C)	--	--	W.Q.	W.Q.	--	A.C.	--	--	W.Q.	W.Q.	--	W.Q.	W.Q.	--	W.Q.	W.Q.
HOT-COLD WORK	REDUCTION, %						--	10	10		--					--	--	--	--	--	--	--	--	--	--
	TEMP., F.	(D)	1250	1250	(D)	1275	--	1420	1420	1240	--	1250	1650	1325	1325	--	--	--	--	--	--	--	--	--	--
FINAL HEAT TREATMENT	TEMP., F.	--	1200	1200	--	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	--	1500	1350	1400	--	1400	1400	1400	(E)
	TIME, HR.	--	10	10	--		4	4	4	4		10	10	4	4	2	--	24	24	16	--	16	16	16	



(A)- CONTOUR
(B)- CHEESE
(C)- AIR COOLED TO 1400 F. AND HELD 24 HR.
(D)- PRESUMABLY HOT-COLD WORKED AFTER HOT FORGING
(E)- 1550 F. 24 HR. PLUS 1300 F., 20 HR.

NOTE:- TEST SPECIMENTS TAKEN NEAR AND RADIALLY TO THE RIM

FIG. 1.—Summarized Tensile Properties at Room Temperature, 1200, 1350, and 1500 F. for Heat-Resisting Alloy Forged Rotor Disks.



ALLOY		TIMKEN 16-25-6					CSA			EME	19-9DL					LOW-CARBON N-155				S-590			S-816		INCONEL-X
DISK NO.		1	2*	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
TYPE OF FORGING		A	A	A	B	A	B	B	B	A	B	A	A	A	A	B	B	B	B	B	B	B	B	B	B
SIZE OF FORGING	DIAMETER, IN.	20	20	20	28	28	20 1/8	20	20	21	19 3/4	20	20	12	12	21	← 14 3/4 →			18	18	18	17	17	22
	THICKNESS, IN.	← 3 1/2 →			4	2 3/8	3 5/16	← 3 1/2 →		4 1/2	3 1/4	← 3 1/2 →		1	1	3 1/4	← 4 3/8 →			3 3/8	3	3	3 5/16		3 1/2
FORGING FINISHING TEMP., F.		NOT REPORTED					1400	1650	1650	1700	1640					1630	1500	1500	1500						
SOLUTION TREATMENT	TEMP., F	--	--	2150	--	--	--	2075	2075	--	--	2150	2150	--	2100	--	--	2200	2200	--	2300	2300	--	2300	2100
	TIME, HR	--	--	2	--	--	--			--	--	2	2	--	1	--	--			--	← 3 1/4 →		--	2 1/2	4
	COOLING	--	--	W.Q.	--	--	--	A.C.	(C)	--	--	W.Q.	W.Q.	--	A.C.	--	--	W.Q.	W.Q.	--	W.Q.	W.Q.	--	W.Q.	W.Q.
HOT-COLD WORK	REDUCTION, %						--	10	10		--					--	--	--	--	--	--	--	--	--	--
	TEMP., F	(D)	1250	1250	(D)	1275	--	1420	1420	1240	--	1250	1650	1325	1325	--	--	--	--	--	--	--	--	--	--
FINAL HEAT TREATMENT	TEMP., F	--	1200	1200	--	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	--	1500	1350	1400	--	1400	1400	1400	(E)
	TIME, HR.	--	10	10	--		4	4	4	4		10	10	4	4	2	--	24	24	16	--	16	16	16	

100-HR. RUPTURE STRENGTHS
1000-HR. RUPTURE STRENGTHS
ELONGATION AT RUPTURE IN 100 HR.
ELONGATION AT RUPTURE IN 1000 HR.
STRESS, PSI.
PER CENT
80 000
70 000
60 000
50 000
40 000
30 000
20 000
10 000
0
60 000
50 000
40 000
30 000
20 000
10 000
0
40
30
20
10
0
40
30
20
10
0
o —— 1200 F.
x ----- 1350 F.
Δ —·—·— 1500 F.

(A) - CONTOUR

(B) - CHEESE

(C) - AIR COOLED TO 1400 F. AND HELD 24 HR.

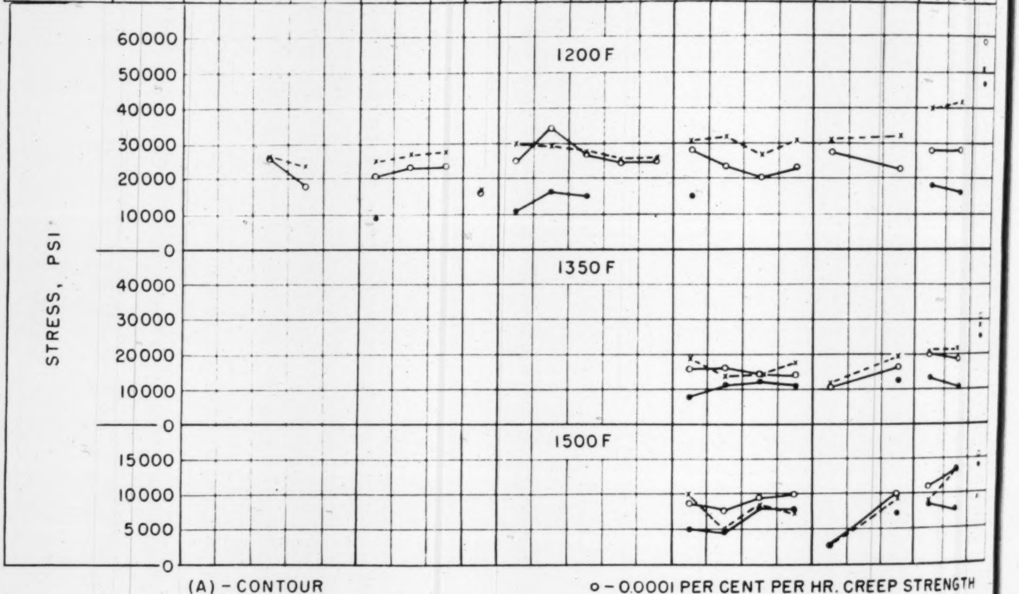
(D) - PRESUMABLY HOT-COLD WORKED AFTER HOT FORGING

(E) - 1550 F. 24 HR. PLUS 1300 F. 20 HR.

NOTE: - TEST SPECIMENTS TAKEN NEAR AND RADIALLY TO THE RIM

FIG. 2.—Summarized Rupture Properties at 1200, 1350, and 1500 F. for Heat-Resisting Alloy Forged Rotor Disks.

ALLOY		TIMKEN 16-25-6					CSA			EME	19-9DL					LOW-CARBON N155				S-590			S-816		INCONEL-
DISK NO.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
TYPE OF FORGING		A	A	A	B	A	B	B	B	A	B	A	A	A	A	B	B	B	B	B	B	B	B	B	B
SIZE OF FORGING	DIAMETER, IN.	20	20	20	28	28	20 1/8	20	20	21	19 3/4	20	20	12	12	21	← 14 3/4 →			18	18	18	17	17	22
	THICKNESS, IN.	← 3 1/2 →			4	2 3/8	3 5/16	← 3 1/2 →		4 1/2	3 1/4	← 3 1/2 →		1	1	3 1/4	← 4 3/8 →			3 3/8	3	3	3 5/16		3 1/2
FORGING FINISHING TEMP., F		NOT REPORTED					1400	1650	1850	1700	1640					1630	1500	1500	1500						
SOLUTION TREATMENT	TEMP., F	--	--	2150	--	--	--	2075	2075	--	--	2150	2150	--	2100	--	--	2200	2200	--	2300	2300	--	2300	2100
	TIME, HR	--	--	2	--	--	--			--	--	2	2	--	1	--	--			--	← 3 1/4 →		--	2 1/2	4
	COOLING	--	--	W.Q.	--	--	--	A.C.	(C)	--	--	W.Q.	W.Q.	--	A.C.	--	--	W.Q.	W.Q.	--	W.Q.	W.Q.	--	W.Q.	W.Q.
HOT-COLD WORK	REDUCTION, %						--	10	10		--					--	--	--	--	--	--	--	--	--	--
	TEMP., F.	(D)	1250	1250	(D)	1275	--	1420	1420	1240	--	1250	1650	1325	1325	--	--	--	--	--	--	--	--	--	--
FINAL HEAT TREATMENT	TEMP., F.	--	1200	1200	--	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	--	1500	1350	1400	--	1400	1400	1400	(E)
	TIME, HR	--	10	10	--		4	4	4	4		10	10	4	4	2	--	24	24	16	--	16	16	16	



(A) - CONTOUR
(B) - CHEESE
(C) - AIR COOLED TO 1400 F AND HELD 24 HR.
(D) - PRESUMABLY HOT-COLD WORKED AFTER HOT FORGING
(E) - 1550 F., 24 HR. PLUS 1300 F., 20 HR

○ - 0.0001 PER CENT PER HR. CREEP STRENGTH
● - 0.00001 PER CENT PER HR. CREEP STRENGTH
x - 10000 HR. RUPTURE STRENGTH

NOTE:- TEST SPECIMENS TAKEN NEAR AND RADIALLY TO THE RIM

FIG. 3.—Summarized Creep Strength and Extrapolated 10,000-hr. Rupture Strengths at 1200, 1350, and 1500 F. for Heat-Resisting Alloy Forged Rotor Disks.

even though the producer prefers to solution treat and age these alloys.

EXPERIMENTAL PROCEDURE

Most of the test work was done on specimens cut from areas of the disks near the rim and taken radially to the rim. The rim area of a turbine rotor disk is subjected to the higher temperatures, and the principal stress occurs in the radial direction.

The specimens used for tension and creep tests were standard 0.505-in. diameter test bars. Rupture tests were made on specimens 0.160 and 0.250 in. in diameter with 1-in. gage lengths.

The test program was chosen to provide the type of data requested by gas-turbine manufacturers and included short-time tension tests at room and elevated temperatures, rupture, and creep tests. Room-temperature tensile data have been included primarily to show the level of properties resulting from the various treatments and in particular to show to some degree the effect of hot-cold work in contour forged disks. Most builders of gas turbines ask for rupture properties as a measure of maximum strength and plastic deformation characteristics of a material. Design data for various amounts of total deformation (elastic plus plastic) for time periods up to 2000 hr. are given for all of the disks. Data for these "design curves" were taken from time-elongation measurements made during rupture and creep tests. Creep tests were made both to obtain data for use in preparing the design curves and to provide the usual creep rate data for those users who are accustomed to estimating performance over prolonged time periods from creep data. In some cases, however, the number of creep tests or their duration was not such as completely to define the creep properties with regard to the stresses producing very low creep rates.

All materials were tested at 1200 F., as this was the temperature indicated to be of greatest interest in current designs of gas turbines, and, considering the compositions of the various materials, also the lowest temperature at which comparisons of high-temperature properties would be particularly significant.

Not all the materials were tested at 1350 and 1500 F. because some of the alloys were either not suitable or were not properly processed for service at these higher temperatures. For instance, the less highly alloyed materials were tested either in the hot-forged condition or as hot-cold worked, both of which conditions usually show lower properties at 1350 and 1500 F. than those produced by solution treatment and aging. The less highly alloyed materials were hot-cold worked primarily to develop high room-temperature yield strength at the hub and to enhance the strength at 1200 F., the estimated operating temperature of the rims of disks of some of the types tested.

RESULTS

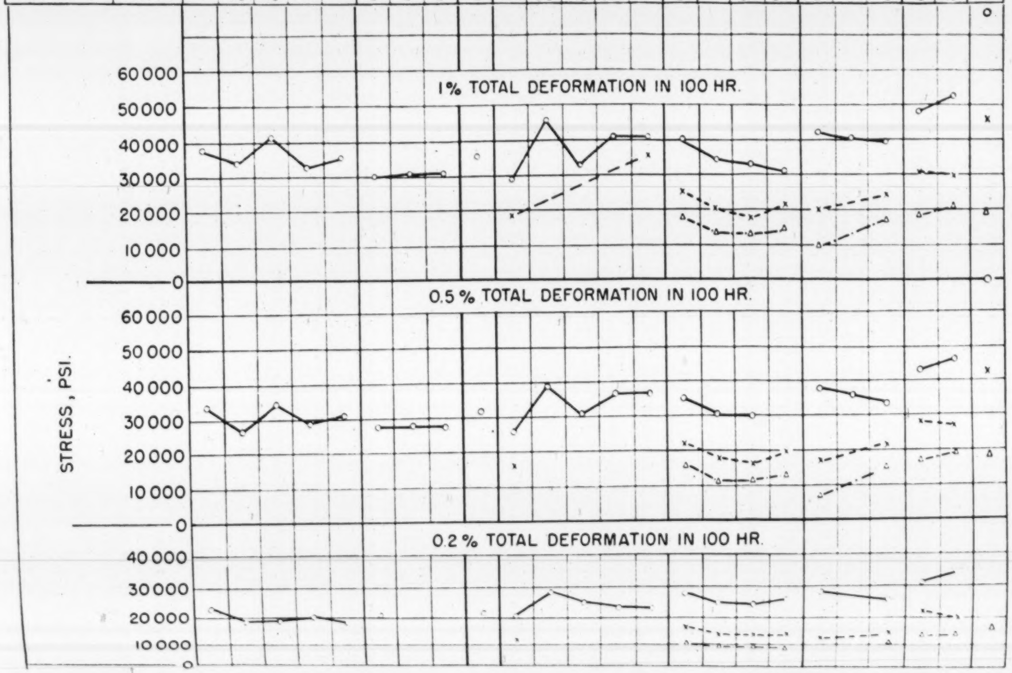
The room-temperature, 1200, 1350, and 1500 F. tensile properties of the disks are summarized in Fig. 1.

The rupture test characteristics at 1200, 1350, and 1500 F. including stresses for rupture in 100 and 1000 hr., and the estimated elongations at rupture are summarized in Fig. 2.

The creep strengths are shown in Fig. 3. In presenting the creep data, the possible danger in extrapolation to prolonged time periods was recognized and accordingly the 0.0001 per cent per hr. creep strengths are compared with the extrapolated stress for rupture in 10,000 hr. in each case.

The stress-time for total deformation characteristics for 0.2, 0.5, and 1.0 per cent total deformation (elastic plus plastic) in 100 and 1000 hr. are summarized in Fig. 4.

ALLOY		TIMKEN 16-25-6					CSA			EME	19-9DL					LOW-CARBON N-155				S-590			S-816		INCONEL-X
DISK NO.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
TYPE OF FORGING		A	A	A	B	A	B	B	B	A	B	A	A	A	A	B	B	B	B	B	B	B	B	B	B
SIZE OF FORGING																									
	DIAMETER, IN.	20	20	20	28	28	20 1/8	20	20	21	19 3/4	20	20	12	12	21	←14 3/4→			18	18	18	17	17	22
	THICKNESS, IN.	←3 1/2→			4	2 3/8	3 5/16	←3 1/2→		4 1/2	3 1/4	←3 1/2→		1	1	3 1/4	←4 3/8→			3 3/8	3	3	3 5/16		3 1/2
FORGING FINISHING TEMP., F		NOT REPORTED					1400	1650	1650	1700	1640					1630	1500	1500	1500						
SOLUTION TREATMENT	TEMP., F	--	--	2150	--	--	--	2075	2075	--	--	2150	2150	--	2100	--	--	2200	2200	--	2300	2300	--	2300	2100
	TIME, HR	--	--	2	--	--	--			--	--	2	2	--	1	--	--			--	←3 1/4→		--	2 1/2	4
	COOLING	--	--	W.Q.	--	--	--	A.C.	(C)	--	--	W.Q.	W.Q.	--	A.C.	--	--	W.Q.	W.Q.	--	W.Q.	W.Q.	--	W.Q.	W.Q.
HOT-COLD WORK	REDUCTION, %						--	10	10		--					--	--	--	--	--	--	--	--	--	--
	TEMP., F.	(D)	1250	1250	(D)	1275	--	1420	1420	1240	--	1250	1650	1325	1325	--	--	--	--	--	--	--	--	--	--
FINAL HEAT TREATMENT	TEMP., F.	--	1200	1200	--	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	1200	--	1500	1350	1400	--	1400	1400	1400	(E)
	TIME, HR.	--	10	10	--		4	4	4	4		10	10	4	4	2	--	24	24	16	--	16	16	16	



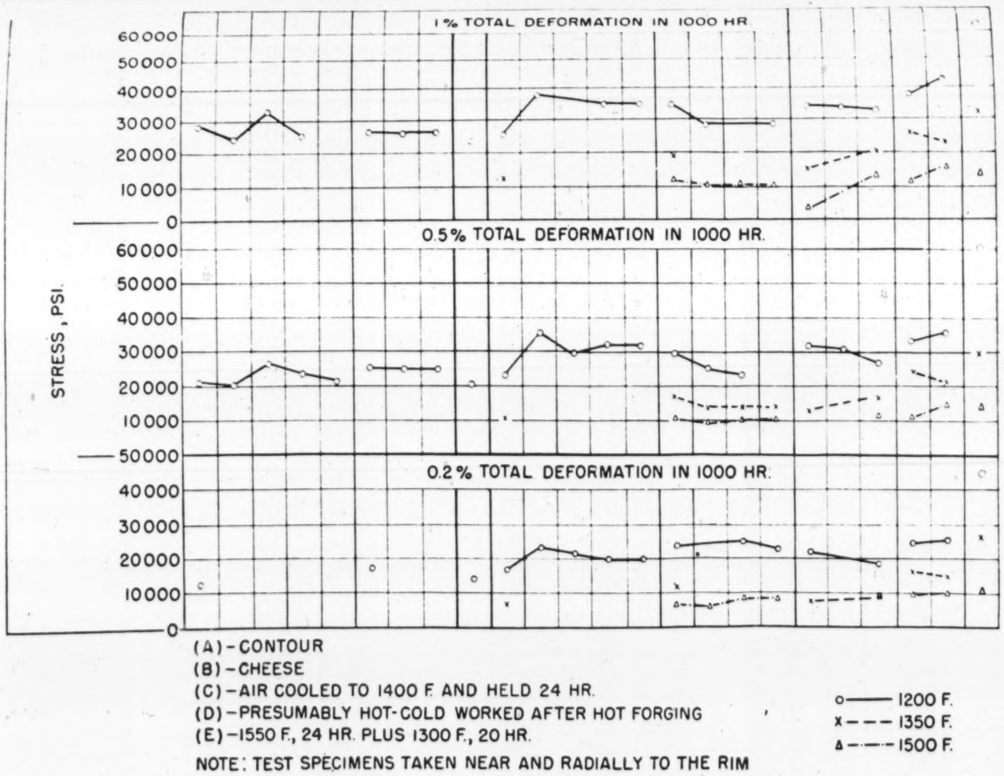


FIG. 4.—Summarized Stress-Time for Total Deformation Strengths at 1200, 1350, and 1500 F. for Heat-Resisting Alloy Forged Rotor Disks.

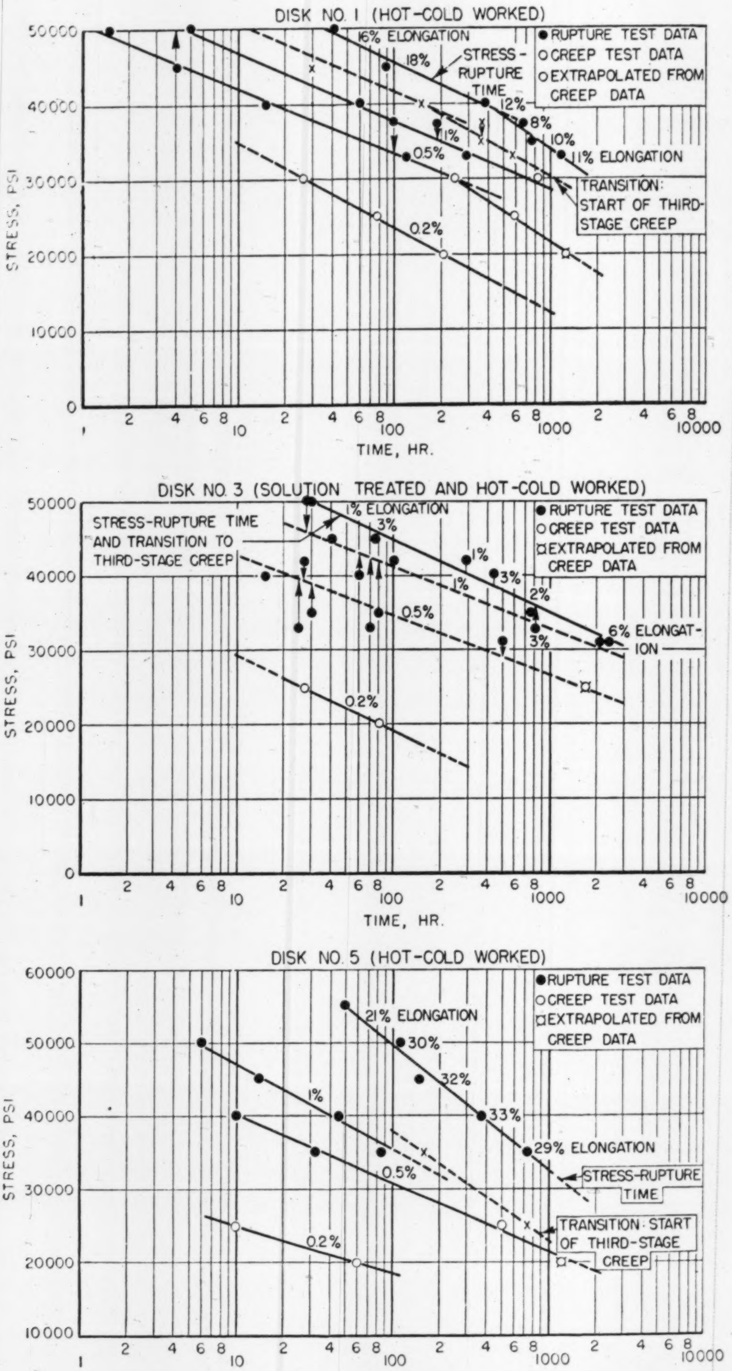


FIG. 5.—Design Data for Timken 16-25-6 Alloy Disks Tested at 1200 F. (For details of processing, see Table II.)

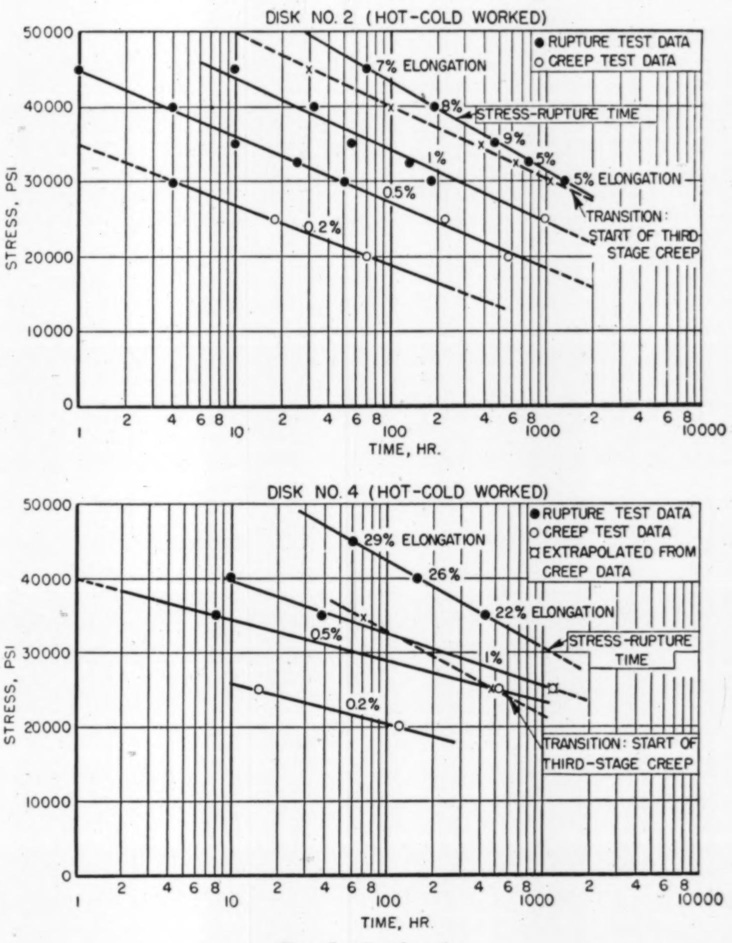


Fig. 5—*Continued*

Both the rupture and deformation characteristics are given in greater detail in the design curves of Figs. 5 through 17.

DISCUSSION OF RESULTS

Properties of the alloys considered in this paper are unquestionably influenced at high temperatures by many variables normally encountered in production. Work on bar stock has indicated that the degree of reduction at temperatures below the normal hot-working temperature, either during hot-forging or during the so-called hot-cold-working operation, has a very potent effect on properties. Temperatures of solution treating and aging (or stress relieving) also exert a pronounced effect on the properties. The relative influence of such treatments varied depending on the alloy, test temperature, time period considered, and on the total deformation (or strain rate) considered.

The properties given in this paper for the various alloys in the form of large disks do not represent true comparisons between alloys in many cases. The less highly alloyed materials were produced under conditions primarily designed to develop high strength at room tempera-

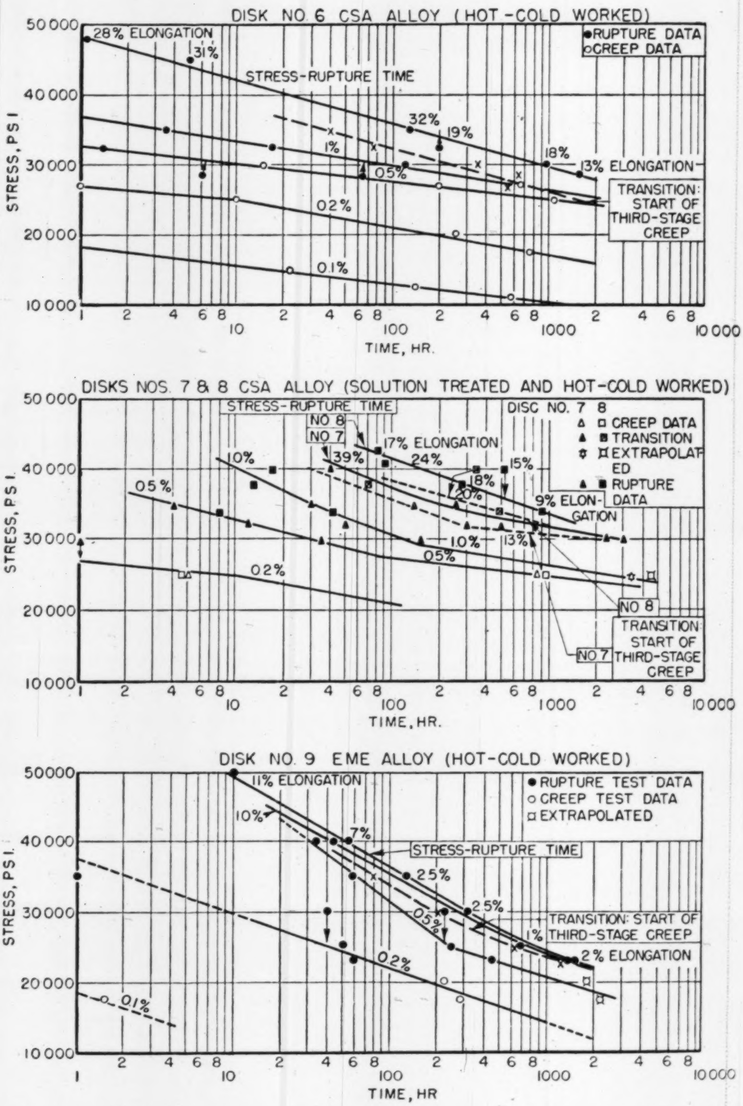


FIG. 6.—Design Data for CSA and EME Alloy Disks Tested at 1200 F. (For details of processing see Table II.)

ture and high rupture strength at 1200 F. The more highly alloyed materials were treated primarily to produce high strength at 1350 and 1500 F. The hot forging or hot-cold working given the less highly alloyed materials is known to be detrimental to the strength of those alloys to some extent at 1350 F. and to a pronounced extent at 1500 F. Likewise the treatments given the more highly alloyed materials are not necessarily the best treatments for high strength at room temperature and 1200 F.

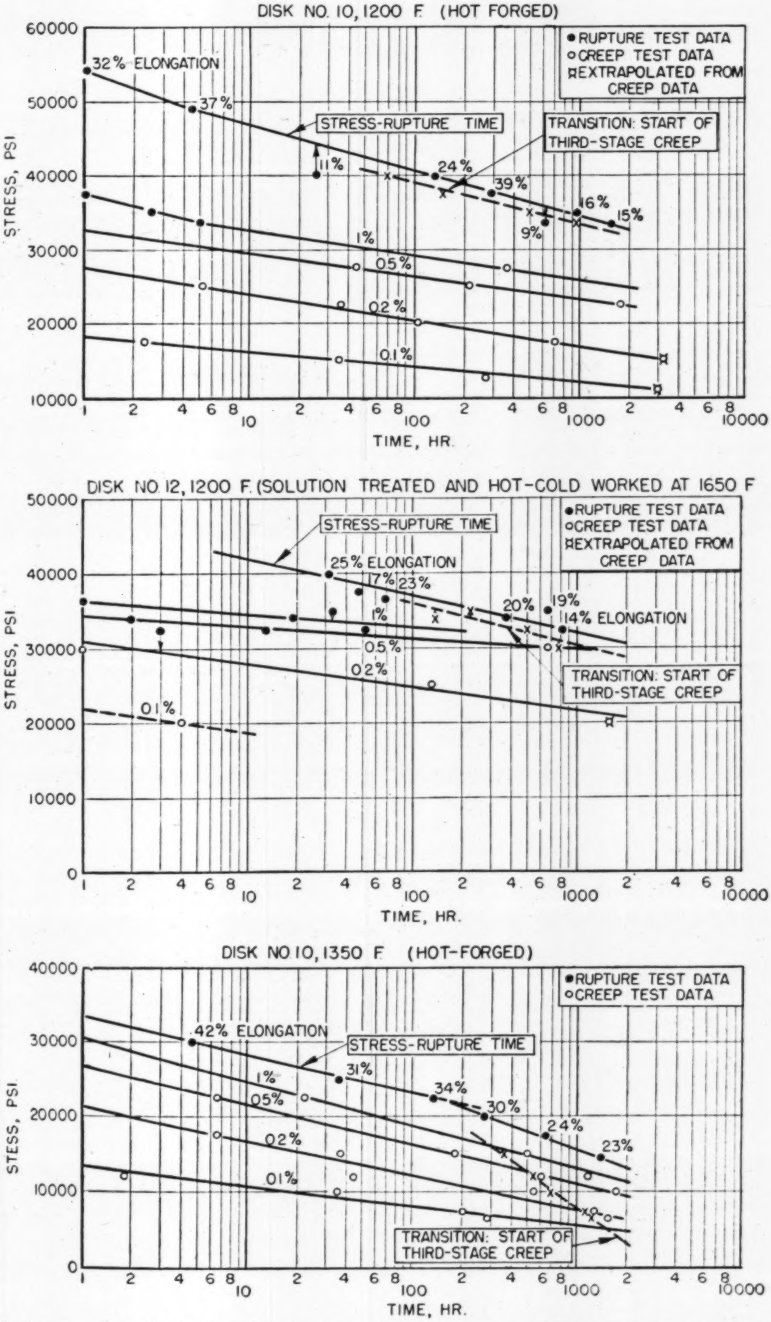


Fig. 7.—Design Data for 19-9 DL Alloy Disks Tested at 1200 and 1350 F. (For details of processing, see Table II.)

(Fig. 7. continued on p. 570.)

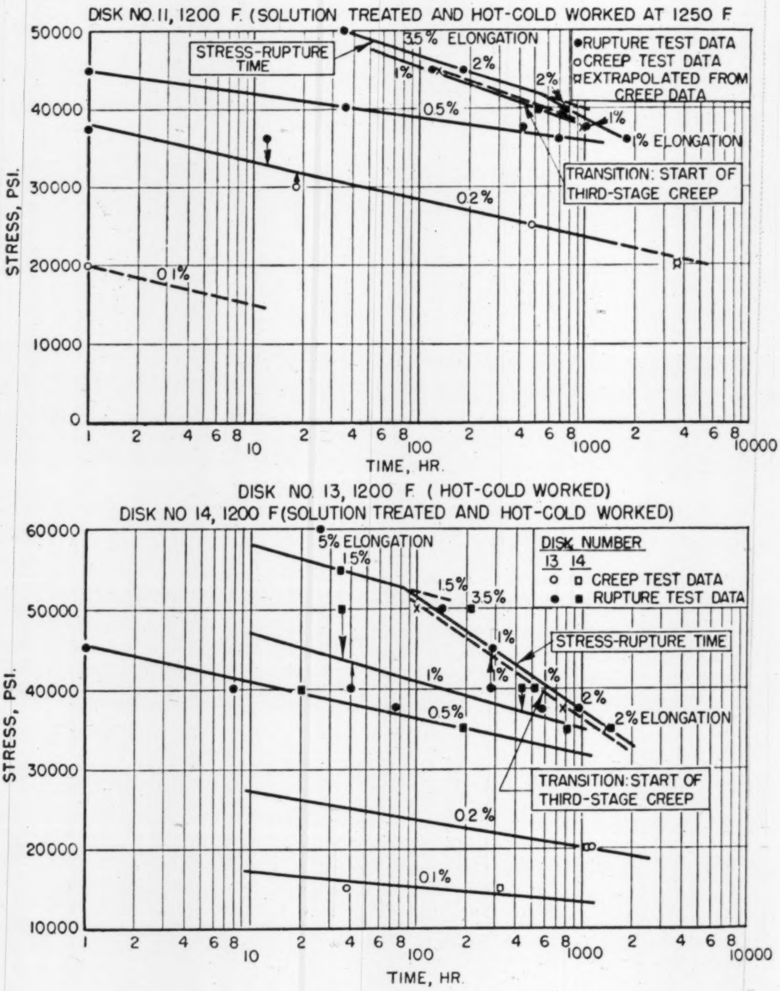


FIG. 7—*Continued*

The general feeling of the authors is that the properties of experimental bar stock of the various alloys have been reproduced in the large forgings to the degree to which the conditions of production have been reproduced. Bar stock properties were reproduced sufficiently well in most cases so that it is reasonably certain that proper adjustment of production procedures would reproduce the high strength of bar stock in all cases. In certain disks, particularly those of EME and CSA alloys, it is reasonably certain that low strength was due to insufficient experience and development work at the time the disks were made to assure that the forging and hot-cold working operations were carried out under the proper conditions to develop the optimum properties of which the alloys were capable.

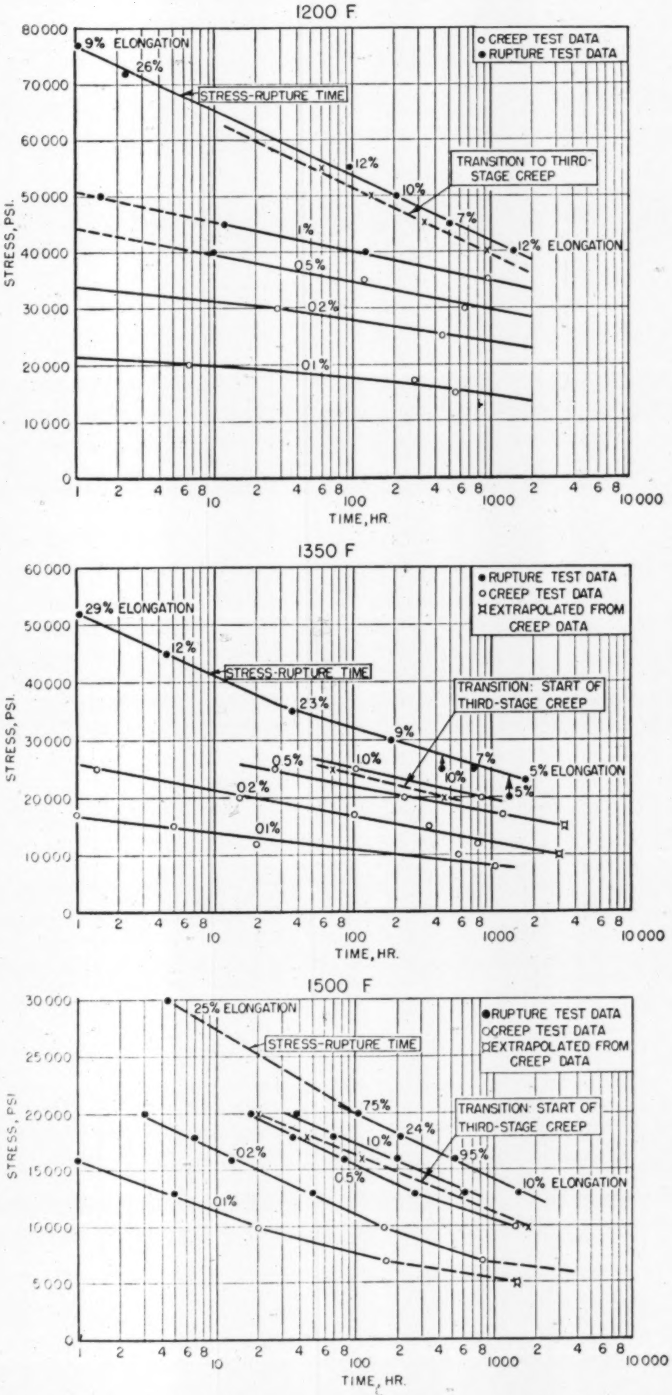


FIG. 8.—Design Data for Low-Carbon N-155 Alloy Forged Disk Tested at 1200, 1350, and 1500 F Disk No. 15 Hot-Forged. (For details of processing, see Table II.)

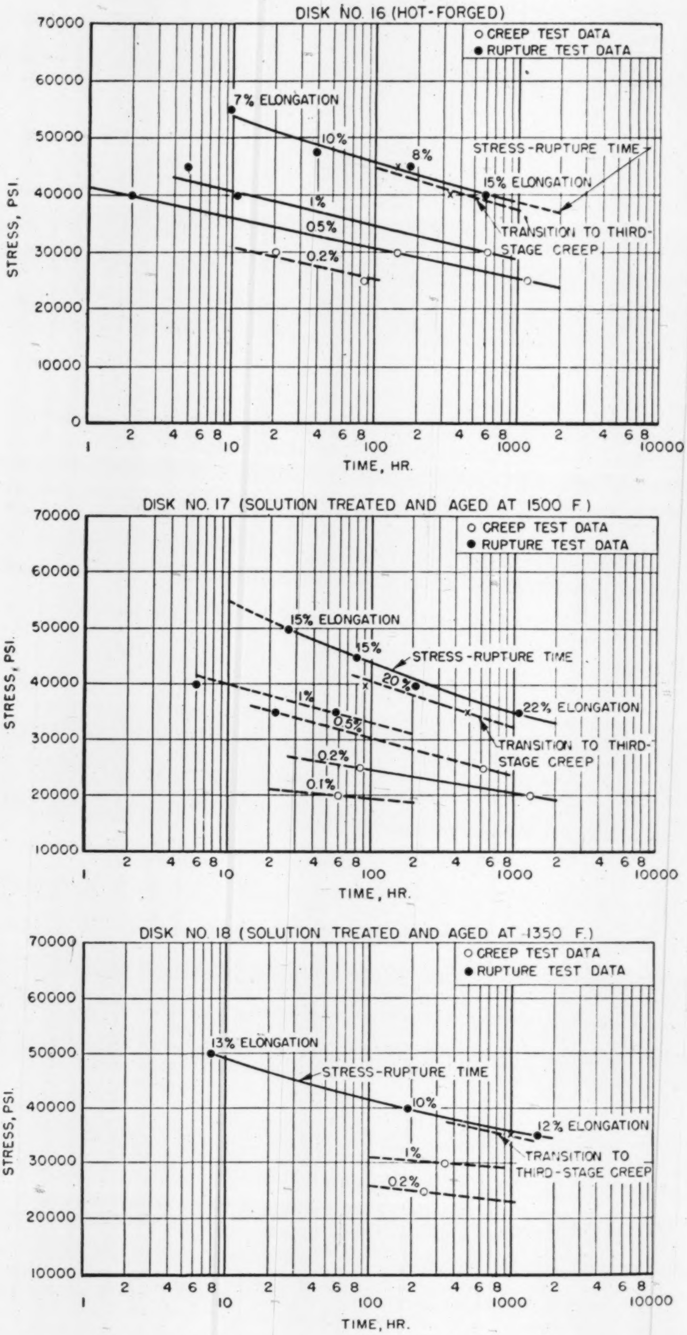


FIG. 9.—Design Data for Low-Carbon N-155 Alloy Forged Disks Tested at 1200 F. (For details of processing, see Table II.)

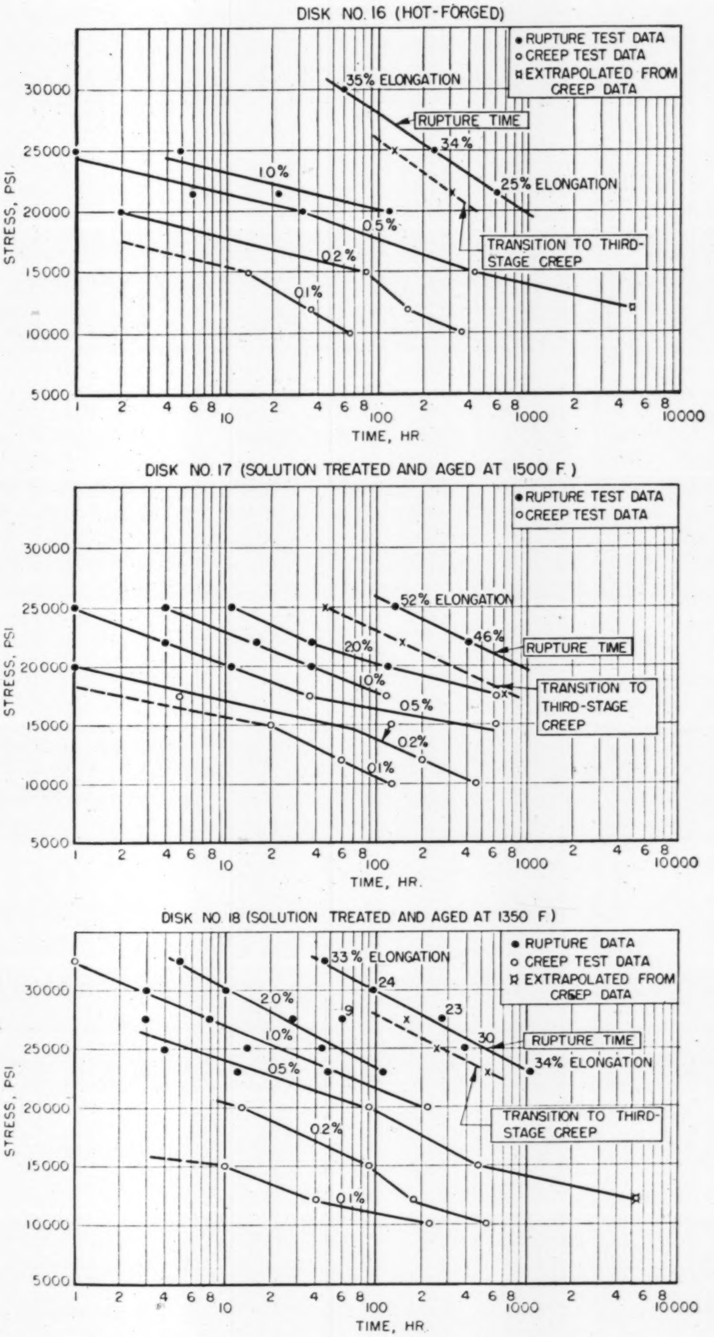


Fig. 10—Design Data for Low-Carbon N-155 Alloy Forged Disks Tested at 1350 F. (For details of processing, see Table II.)

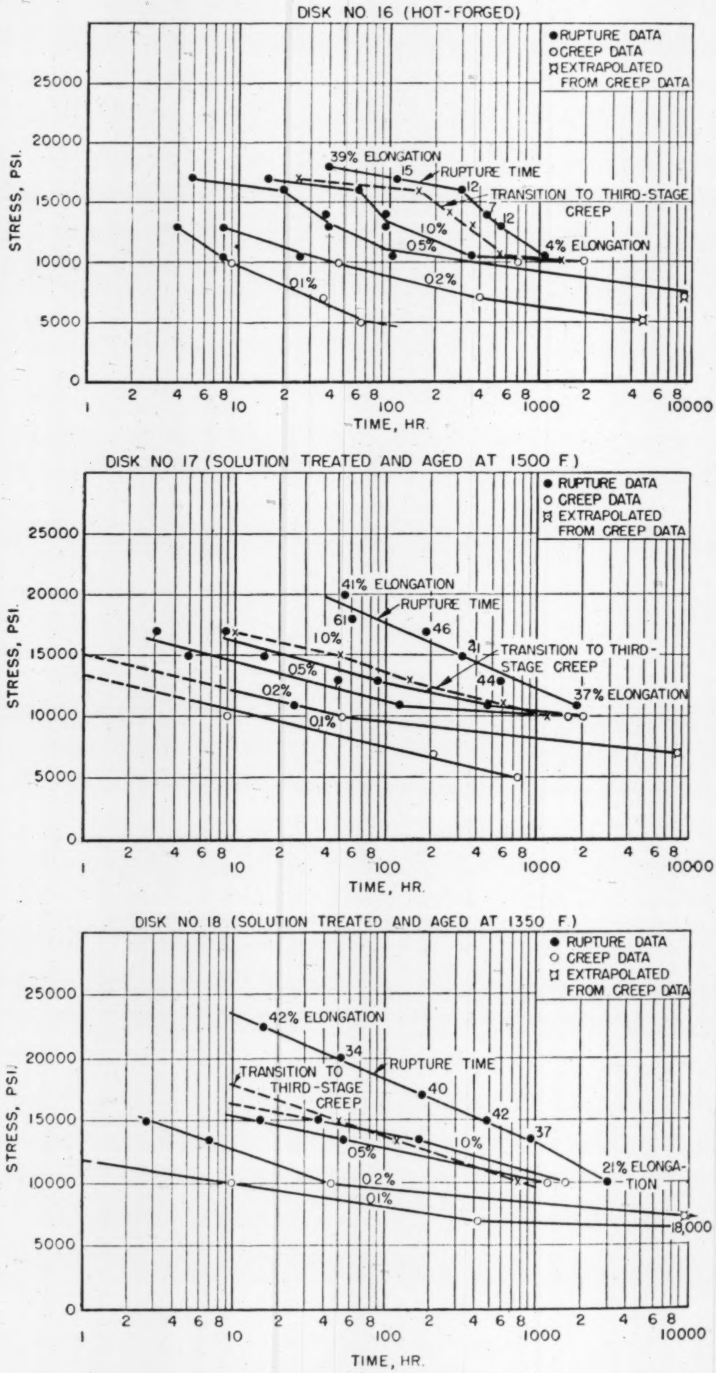


FIG. 11.—Design Data for Low-Carbon N-155 Alloy Forged Disks Tested at 1500 F. (For details of processing, see Table II.)

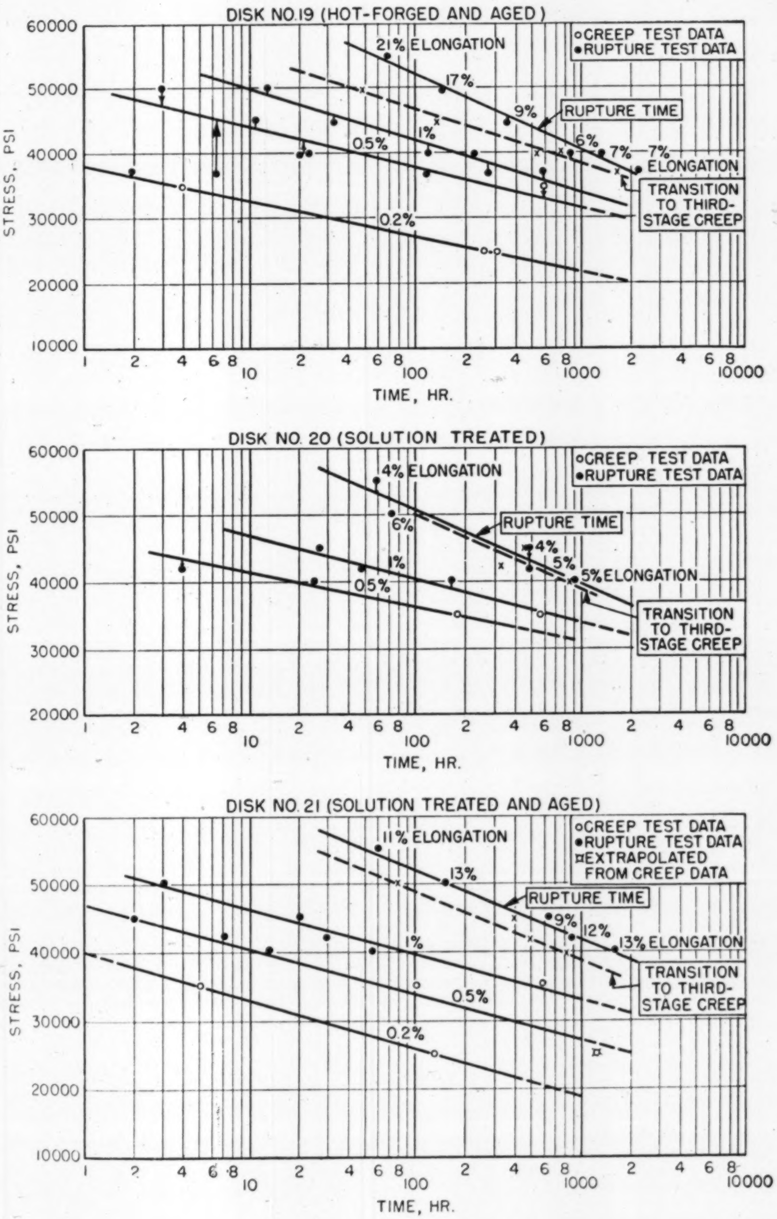


FIG. 12.—Design Data for S-590 Alloy Forged Disks Tested at 1200 F. (For details of processing, see Table II.)

There are a large number of details involved in the production and testing of the disks. In a paper of this type it is impossible to discuss all such details. There are, however, certain generalizations which can be made.

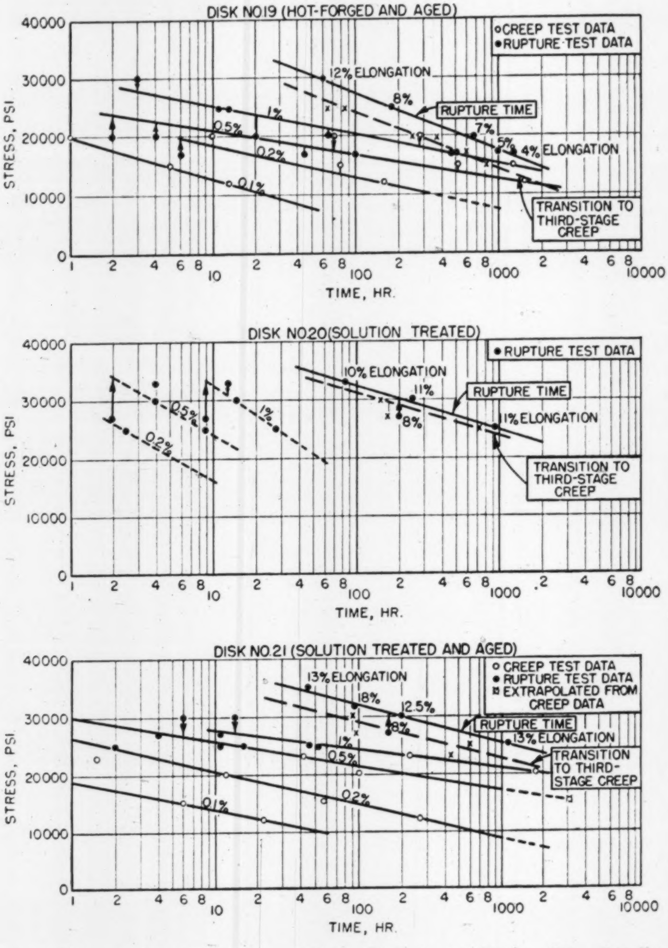


FIG. 13.—**Design Data for S-590 Alloy Forged Disks Tested at 1350 F.** (For details of processing see Table II.)

Tensile Properties:

The tensile properties of the disks of the four lower alloyed materials, Timken 16-25-6, CSA, EME, and 19-9DL, reflect the degree of hot-cold work imparted during forging. There is no reason to believe that the treatments given the disks of the various alloys were sufficiently uniform so that the strengths necessarily reflect the effect of chemical composition. Their tensile and yield strengths are all considerably higher than the same alloys would have after a solution treatment with or without aging.

Disk 3 of Timken 16-25-6 and disk 11 of 19-9DL alloy were produced under reasonably similar conditions and have no significant difference in strength. The data for disk 3 suggest that a solution treatment prior to hot-cold work results in higher strength and lower ductility than obtained by hot-cold working as-forged material. However, more data are needed to verify whether this trend was due to the heat treatment and not to variation in the hot-cold-working operation.

carbon N-155, S-590, and S-816 alloys, the tensile and yield strengths at room temperature were higher for the hot-forged material than for solution-treated material. This superiority, although decreased, was maintained at 1200 F. At 1350 F. and 1500 F., the tensile and yield strengths were quite similar for the as-forged and the heat-treated conditions. The tensile strengths of the low-carbon N-155, S-590, S-816, and Inconel-X disks decreased with temperature increase from 1200 to 1500 F. Much smaller variations with temperature were observed for the yield strengths; in fact, the 0.2 per cent yield strengths for the solution-treated and aged disks 17 and 18 increased rather than decreased with the test temperature.

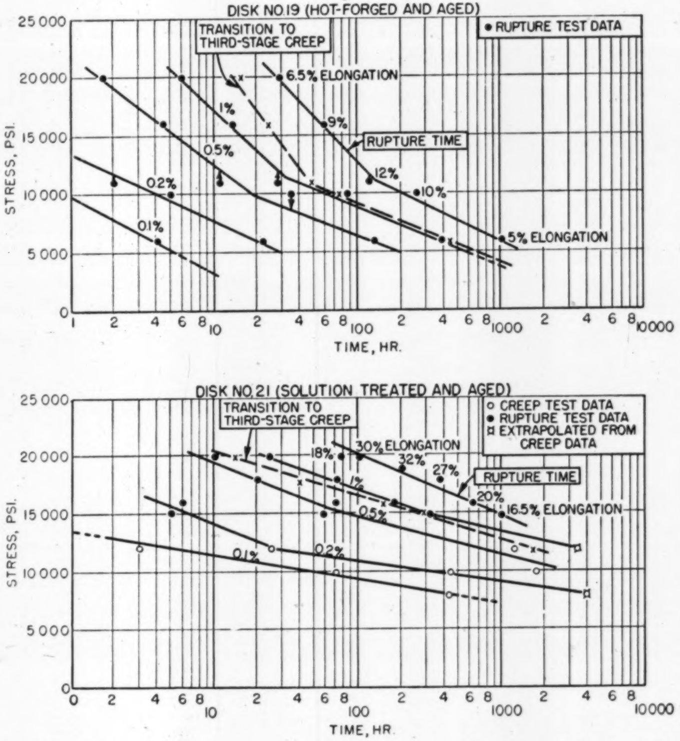


Fig. 14.—Design Data for S-590 Alloy Forged Disks Tested at 1500 F. (For details of processing, see Table II.)

The data for disk 12 of 19-9DL indicate that lower strength and higher ductility result from hot-cold working at temperatures (1650 F.) above about 1200 to 1400 F. The lower strengths of CSA disks 7 and 8 probably reflect the effect of both smaller amounts of hot-cold work and somewhat higher hot-cold-working temperature than some of the other disks.

For the higher alloyed materials low-

For S-590 and S-816 alloys, room-temperature elongations were lower for as-forged material than for solution-treated and aged alloys. For S-590, elongations were much lower at 1200 and 1350 F. when only solution-treated, as compared with the alloy solution-treated and aged.

The solution-treated and aged Inconel-X maintained high levels of tensile properties over the entire temperature range, and was exceeded only by S-816 alloy in tensile strength in the temperature range 1200–1500 F. and in yield strength at 1500 F. The elongation value for Inconel-X was considerably lower at 1200 F. than at room temperature and at 1500 F., and was at a minimum at 1350 F.

It will be noted that the yield strengths of the low-alloyed disks in the hot-cold-worked condition compared quite favorably at room temperature and 1200 F. with those of the higher alloyed materials, except for Inconel-X which had considerably higher strength.

The greater difference in strength between the forged-and-aged and solution-treated disks of S-590 alloy as compared with the small difference observed for S-816 alloy quite certainly results from differences in the forging procedures used for these two alloy disks rather than from differences between their chemical compositions.

Rupture Properties:

The summarized rupture data in Fig. 2 suggest several significant trends.

The five disks of Timken alloy show only minor variation in rupture strengths, particularly at 1000 hr., from differences in processing. There were wide differences in elongation, with the larger disks having the highest elongation and the disk solution-treated prior to hot-cold work the lowest.

Quite large differences in chemical composition and processing did not significantly change the rupture strengths of CSA disks. The rupture strengths at 100 hrs. were less than those of Timken 16-25-6 disks, and both were similar at 1000 hr., as would be anticipated from the probably smaller amount and higher temperature of hot-cold work on the CSA disks. The elongations of the CSA alloy disks 7 and 8 were somewhat higher than might be expected for solution-treated and hot-cold-worked disks, probably both because the reduction was carried out at a higher temperature than for other disks and because 10 per cent reduction was probably less than usual.

The rupture strengths of the EME disk are considered to be a good example of an early experimentally produced disk which had considerably lower rupture strength than had previously been indicated for the alloy by hot-cold-worked bar stock, because of lack of knowledge of the proper fabricating conditions for large disks. Similar properties could very easily result for the other alloys, and actually probably accounts for the somewhat low strength of the CSA disks. It is to be expected that suitable commercial procedures could be developed which would reproduce the properties of bar stock as well as for the other alloys.

The rupture strengths of the 19-9DL disks at 1200 F. reflect the differences in processing in that the severely hot-cold-worked disks 13 and 14 had the highest 100-hr. strength, disk 11 somewhat lower strength, while the disk 12 hot-cold worked at 1650 F. and the as-forged disk 10 were the weakest.

It will be noted that the rupture strengths of Timken 16-25-6 alloy do not show appreciable differences even though there were wide differences in tensile and yield strength shown in Fig. 1. In general the rupture and yield strengths of CSA and 19-9DL alloys both follow

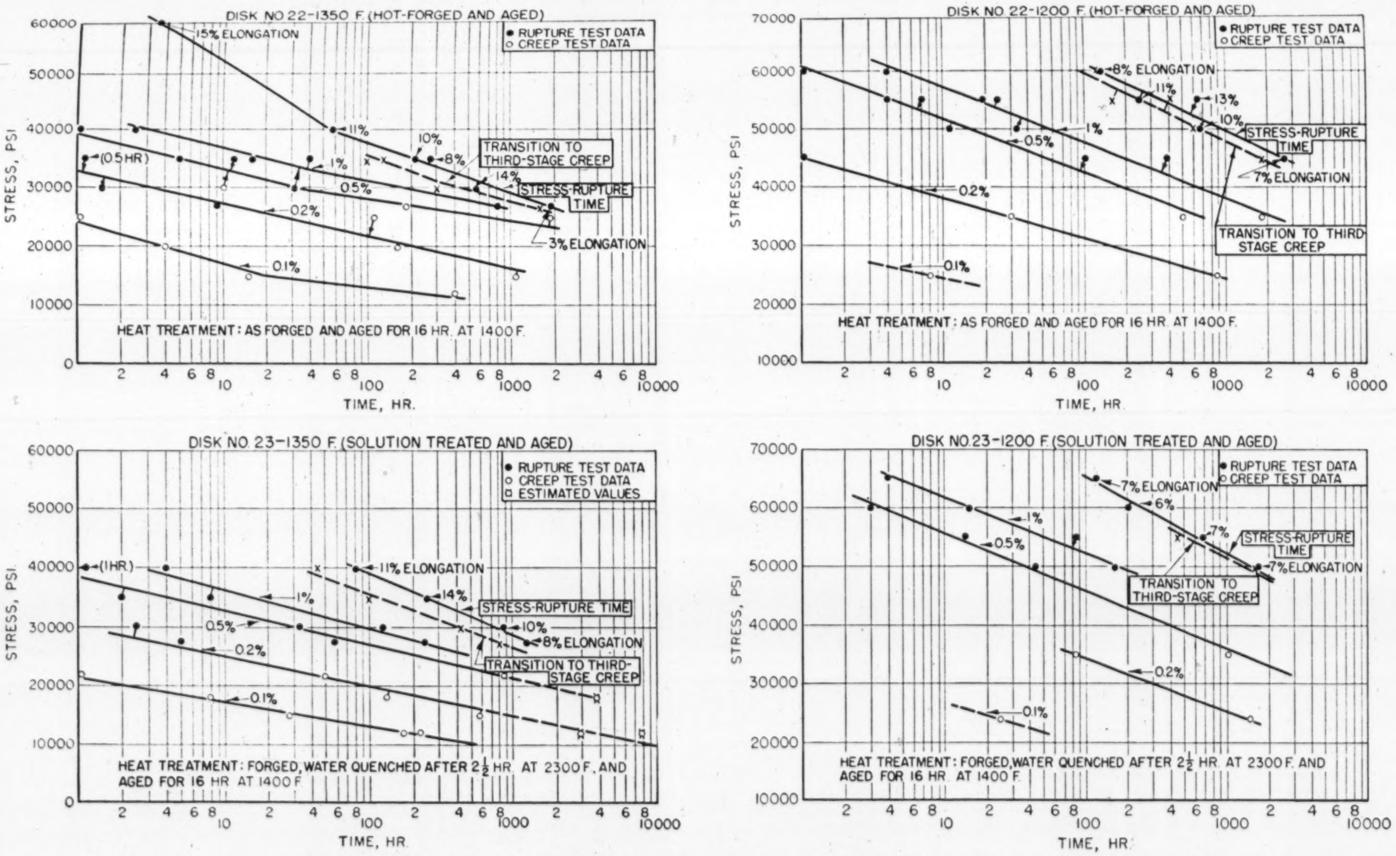


FIG. 15.—Design Data for S-816 Alloy Forged Disks Tested at 1200 and 1350 F. (For details of processing, see Table II.)

the same trend. The EME disk did not have high rupture strength even though it had high tensile and yield strength.

There is relatively little difference in rupture strength for 1000 hr. at 1200 F. for three of the less highly alloyed disks: Timken 16-25-6, CSA, and 19-9DL. Limited data at 1350 F. show relatively little difference in rupture strength for CSA and 19-9DL disks.

The data for Timken 16-25-6 and 19-9DL alloys suggest that solution-treated and hot-cold-worked disks have higher rupture strength at 100 hr. than when hot-forged disks are hot-cold worked. It is not considered, however, that the available data adequately substantiate this trend. There is no question but that equal strengths could be developed by hot-cold working as-forged disks provided the forging conditions are suitably controlled. Actually, however, forging conditions may be expected to vary and cause variation in rupture strength. In fact, there is a strong possibility that the unexpected low strength of the EME disk was due to forging conditions which adversely affected the rupture strength, and that similar situations could occur in the other alloys.

Elongation values in rupture tests are quite erratic for the less highly alloyed materials. In general they show that the as-forged disks had good elongation, as-forged and hot-cold-worked disks may have either high or very low elongation, and that solution-treated and hot-cold-worked disks will have very low elongation unless the temperature of hot-cold working is above about 1400 F. The elongations of the solution-treated and hot-cold worked CSA alloy disks were not so low as might be expected for such treatments because the reduction was only 10 per cent and the temperature of working was relatively high.

The solution-treated and aged disks of low-carbon N-155 (disks 17 and 18) showed slightly lower rupture strengths at 1200 F. as compared with the as-forged disks. The two as-forged disks varied also, which is another instance of the effect of different forging procedures. The rupture strengths at 1350 and 1500 F. are quite similar for all four disks regardless of fabrication procedure or heat treatment. Considerable reductions of rupture strength occurred as the temperature was increased from 1200 to 1350 and 1500 F. This reduction in rupture strength with temperature is in direct contrast to the uniformity in yield strength and would certainly not be predicted from the closely agreeing 0.2 per cent yield strengths for those materials at 1200, 1350, and 1500 F., as indicated in Fig. 1.

Elongations at rupture for the low-carbon N-155 disks varied considerably from disk to disk, with the lower values in general for the hot-forged disks. The elongation data show, as would be expected, that aging at 1500 F. results in higher elongation than aging at 1350 F.

For the S-590 alloy, the rupture strengths at 1200 F. were quite similar whether the disks were as-forged or solution-treated and aged. The superior yield strength (0.2 per cent) of the as-forged disk at 1200 F. was not reflected in its rupture strength at 1200 F. The quenched-and-aged material had better rupture strengths at 1350 and 1500 F. than the as-forged S-590, with the superiority greater at the highest temperature. The data showed surprisingly little effect of aging on the rupture strength at 1200 and 1350 F.

Elongations at rupture of the S-590 alloy tended to be higher as solution-treated and aged than as-forged. The omission of the aging treatment resulted in lower elongation at 1200 F.

The rupture strengths of as-forged

S-590 alloy were similar to those of as-forged low-carbon N-155 at 1200 F. but were considerably lower at 1350 and 1500 F. The rupture strengths of solution-treated and aged S-590 were higher at 1200 F. than for heat-treated low-carbon N-155 but were quite similar at 1350 and 1500 F.

The rupture strengths of as-forged-and-aged, and the solution-treated and aged S-816 alloy were quite similar at each of the test temperatures of 1200, 1350, and 1500 F. Except for Inconel-X, the S-816 alloy showed the highest rupture strengths at 1200 and 1350 F. At 1500 F., as-forged S-816 was quite similar to low-carbon N-155 but was definitely superior to as-forged S-590 alloy. In the solution-treated and-aged condition at 1500 F., S-816 alloy was slightly superior in rupture strength to both low-carbon N-155 and S-590 alloys.

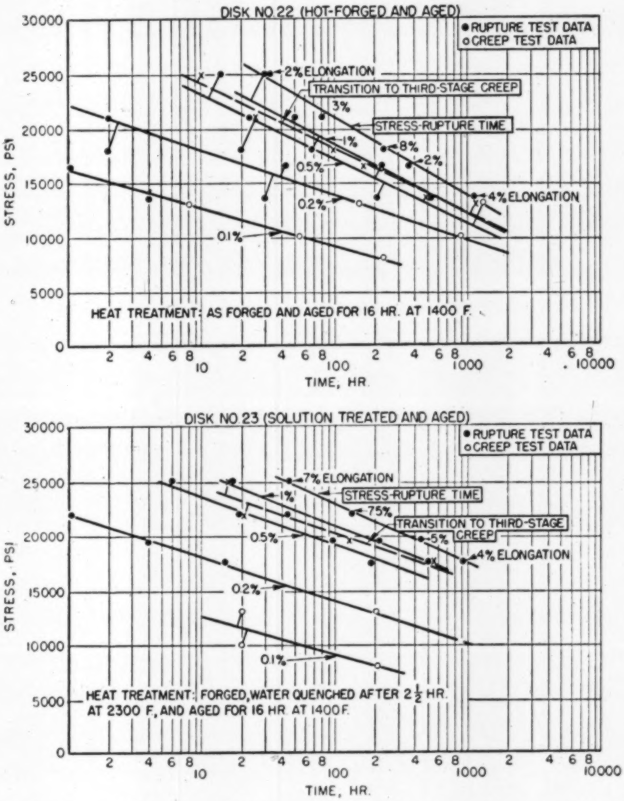


Fig. 16.—Design Data for S-816 Alloy Disks Tested at 1500 F. (For details of processing, see Table II.)

Elongations at rupture for both S-816 alloy disks were consistently low with the lowest values at 1500 F.

Inconel-X had the highest rupture strengths of any of the alloys tested at 1200 and 1350 F. However, the strengths at 1500 F. for both 100 and 1000 hr. fall in the same range as for low-carbon N-155, S-590, and S-819 alloys. The elongations at rupture at

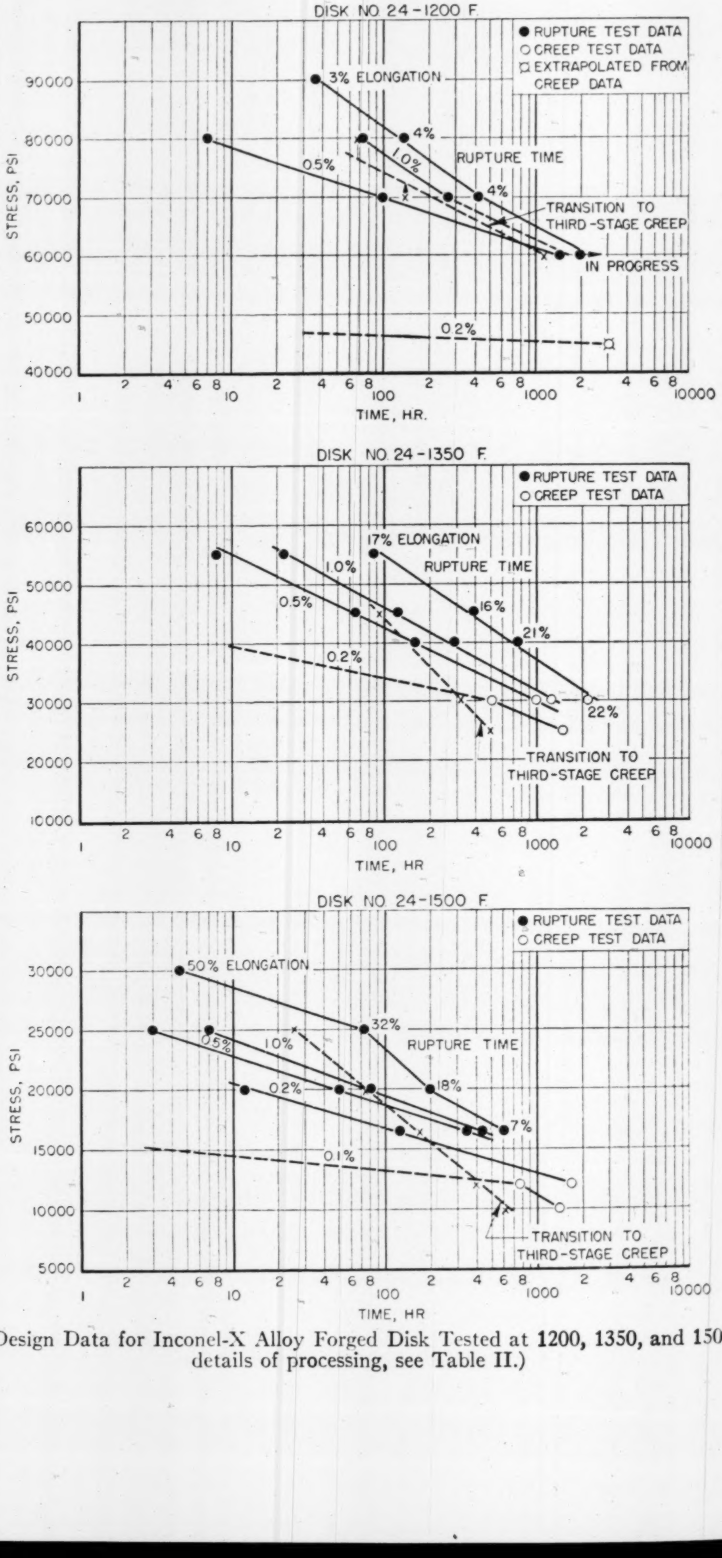


FIG. 17.—Design Data for Inconel-X Alloy Forged Disk Tested at 1200, 1350, and 1500 F. (For details of processing, see Table II.)

1200 F. were somewhat lower than for the other highly alloyed materials, but not so low as those of hot-cold worked alloys of much lower strength. The elongations at rupture in 1000 hr. at 1350 F. were higher than at either 1200 or 1500 F. At 1500 F. the elongation at rupture decreased rapidly with increase in rupture time, decreasing from 32 per cent at 74 hr. to 7 per cent at 604 hr. Additonal important characteristics of Inconel-X will be shown when discussing the creep strengths and the design curves.

In general the rupture data indicated that the differences in rupture strength resulting from different processing procedures for one alloy or between disks made from different alloys tend to decrease as the time period considered and as the testing temperatures were increased. There were no great differences in the rupture strengths of the various disks for 1000 hr. at 1200 F., except for the higher strengths of S-816 and Inconel-X. At 1350 F., the less highly alloyed hot-cold-worked disks were definitely inferior and would have compared even less favorably if tests had been made at 1500 F. At 1350 F., S-816, and to a greater extent Inconel-X, showed higher strength than low-carbon N-155 and S-590, but the degree of superiority was less at 1000 hr. than at 100 hr. At 1500 F., there was practically no difference in strength between these alloys.

Creep Strength:

Figure 3 shows the creep strengths for 0.0001 and 0.00001 per cent per hour and, for purposes of comparison, the extrapolated rupture strengths for 10,000 hr. rupture. On the less highly alloyed materials, Timken 16-25-6, CSA, EME, and 19-9DL, tests were made only at 1200 F., and even at this temperature tests were not made on disks of all of these alloys at sufficiently low stresses to obtain creep rates of 0.0001 and 0.00001 per cent per hour.

Before consideration is given to the actual creep strengths of the various disks or to their relative creep strengths, the important fact to note is that in the majority of cases the 0.0001 per cent per hour creep strengths are not much lower than the estimated 10,000-hr. rupture strengths. In fact, in several cases, notably disk 11 of 19-9DL alloy at 1200 F., Inconel-X at all three temperatures, and the other highly alloyed materials at 1350 and 1500 F., the rupture strengths are either quite similar to or actually less than this creep strength. In some cases, the extrapolated 10,000-hr. rupture strengths were even less than the 0.00001 per cent per hour creep strengths.

These relative rupture and creep strengths indicate that the common practice of extrapolating the creep strengths to indicate that rates of 0.0001 and 0.00001 per cent per hour are equivalent respectively to 1 per cent creep in 10,000 and 100,000 hr. can be seriously in error. In the extreme cases, this extrapolation was so unreliable that actual failure would occur prior to the time periods commonly used in extrapolating creep strengths. In so far as can be determined from the data, this condition exists because the increasing creep rates of so-called third-stage creep occur at deformations considerably less than 1 per cent and in time periods much shorter than 10,000 hr. rather than because failure would occur at less than 1 per cent deformation.

The creep strengths at 1200 F. in general followed the same trends as the rupture strengths. There was no trend for pronounced superiority of any of the alloys except Inconel-X. In the less highly alloyed materials solution treatments followed by hot-cold work at about 1200 F. developed high creep strengths, but they were very close to or above the rupture strengths. Heat-treated low-carbon N-155 and S-590 both tended to show lower creep strength than forged-and-aged disks and a wider separation between rupture and creep strengths. There was no difference between the forged and the solution-treated and aged disks of S-816 alloy. The data for disks 17 and 18 of low-carbon N-155 alloy suggest that better creep strength results from aging at 1350 F. than at 1500 F.

Only low-carbon N-155, S-590, S-816, and Inconel-X alloys were tested at 1350 F. and 1500 F. at sufficiently low stresses to determine their creep strengths. At these temperatures the solution-treated and aged disks developed creep strengths equal to or higher than the as-forged disks. Inconel-X maintained a higher creep strength at these temperatures, but the degree of difference was much less than at 1200 F. Differing from the relation observed at 1200 F., 10,000-hr. rupture strengths at 1350 and 1500 F. for these alloys were very close to or were a little below the creep strengths for a rate of 0.0001 per cent per hour.

Inconel-X alloy behaved quite differently from the other alloys in the creep tests. At the stresses producing low creep rates, there was hardly any so-called "first-stage" creep and the minimum creep rate occurred in the first several hundred hours of the tests. The transition point, or time at which the creep rate begins to increase, occurred early in the test period as is indicated in the design curves of Fig. 17. This tendency was not so marked at 1200 F. but became increasingly evident as the test temperature was raised. All of the other alloys had pronounced periods of decreasing creep rate or first-stage creep before reaching a considerable period of approximately constant and minimum creep rate and finally entering third-stage creep. In fact, at 1200, 1350, and 1500 F., most of the solution-treated and aged disk specimens and the as-forged specimens of the highly alloyed materials did not reach their minimum creep rates during 2000-hr. creep tests at the lower stresses.

Stress-Time for Total Deformation Characteristics:

Figure 4 shows the stresses for 0.2, 0.5, and 1.0 per cent total deformations at 100 and 1000 hr. for the various disk materials tested at 1200, 1350, and 1500 F. as scaled from the design curves shown in Figs. 5 through 17.

The trends indicated follow more closely those of the rupture strengths than those of the short-time tensile properties. Differences resulting from variations in fabrication procedure or heat treatment are less marked at the lower deformation values. Large variations in elongation at rupture are not reflected in these data for stresses producing total deformation of 1 per cent or less for time periods of 100 and 1000 hr., suggesting that for service applications involving total deformations up to this magitude, rupture elongation is not a significant factor.

Design Data:

Design data for the various disk materials are shown in Figs. 5 to 17 as derived from both rupture and creep data. These figures present the relationships between stress and time for rupture, transition point (start of third-stage creep), and total deformations (elastic plus plastic) of 0.1, 0.2, 0.5, and 1.0 per cent. The total deformation includes both the initial deformation (elastic plus plastic) which occurred upon application of the load, and the subsequent creep. The figures adjacent to the individual points on the stress rupture-time curves indicate the elongations at rupture.

The design data serve to relate on one chart the actual deformation produced by a stress and during a time period of interest. The relationship of stress to the actual time for rupture and the total elongation at rupture is clearly evident from the stress rupture-time curve. The transition curve indicates when rapid elongation or third-stage creep prior to rupture starts to occur and the relationship of this characteristic to total deformation and rupture properties.

Figure 5 shows the design data for the five Timken 16-25-6 disks tested, and Fig. 6 shows the design data for the CSA disks and the EME disk tested at 1200 F.

Figure 7 shows the design data for the 19-9DL alloy disks tested at 1200 F. and the one disk tested at 1350 F. The low elongation at rupture of the hot-cold-worked disks 11, 13, and 14 was not accompanied by any significant change in the relationships at deformations of 1 per cent or less; the design data curves are all of the same general type.

The design data for disk 10 tested at 1350 F. showed low strength for 19-9DL alloy at 1350 F., but of even more importance, the location of the transition point falling as low as 0.2 per cent deformation at 1000 hr. clearly indicated that 19-9DL alloy in this condition should not be used at temperatures as high as 1350 F. It should be recognized that proper solution and aging treatments will develop better properties at 1350 F. than those shown by this as-forged disk.

Figure 8 shows the design data for the low-carbon N-155 disk 15 tested in the as-forged condition, and Figs. 9, 10, and 11 show the data for disks 16, 17, and 18 of this alloy tested in the as-forged, and the solution-treated-and-aged conditions. In Fig. 8 the excellent strength of disk 15 at 1200 F. is shown by the slight slopes of the deformation lines. The increasing slopes at the higher temperatures indicated the decrease in strength with temperature increase. At the higher temperatures, the transition point occurred at lower deformations. No appreciable differences between disks are shown in Fig. 9. Figures 10 and 11 indicate that elongations at rupture were much greater at 1350 F. than at 1200 F. or 1500 F. for the as-forged disk, and greater than at 1200 F., but not so great as at 1500 F., for the solution-treated-and-aged disks.

Figure 12 shows the design data for S-590 alloy at 1200 F. Noteworthy indications are that the ductility at rupture was maintained even at the long rupture times, and that the transition to third-stage creep occurs at total deformations in excess of 1 per cent.

Figure 13 shows the design data for S-590 alloy at 1350 F. The rupture elongation at 1350 F. remained practically constant over the range of rupture testing times. These data clearly show the desirability of solution-treating and aging of this alloy for 1350 F. service. That aging is necessary is clearly indicated by the rapid deformation in the early stages for the alloy tested as solution-treated and without aging.

Figure 14 shows the design data for S-590 alloy at 1500 F. The as-forged alloy had very poor load-carrying ability as compared with the solution-treated-and-aged condition. Ductilities at rupture were high at this temperature also. Transition occurred at lower total deformations than at the lower test temperatures.

Figures 15 and 16 show the design data for S-816 at 1200 F. and at 1350 and 1500 F. Elongation at rupture tended to be lower at 1350 and 1500 F. than at

1200 F., and the transition point occurred at a lower total deformation at the two higher test temperatures.

The important indications of design data for Inconel-X at 1200, 1350, and 1500 F. in Fig. 17 are that while this alloy had high stresses for total deformations up to 1 per cent for time periods up to 1000 hr., the transition points occurred at very low total deformations and early in the test period. This situation presents a warning with regard to the dangers of extrapolation of the data to longer time periods than those for which the tests were run. It also suggests the possibility that, at stresses sufficiently low to keep below the transition point, for long-time service Inconel-X may be little, if at all, superior to some of the other alloys which are definitely inferior at higher stresses and over testing periods of up to 1000 hr.

SUMMARY AND CONCLUSIONS

The sources of supply, compositions, fabrication procedures, and heat treatments used for 24 disks of 8 different heat-resisting alloys are given. Some of the disks were tested as hot forged, some as hot-cold worked, and others as solution treated or solution treated and aged. Most of the test work was done on specimens cut from areas of the disks near the rims and taken radially to the rims. Their short-time tensile, rupture, creep, and stress-time for total deformation characteristics were determined at temperatures of 1200, 1350, and 1500 F.

The relative strengths of the materials are dependent, in addition to alloy content, upon the processing and heat treatments used, the time period considered, the criterion used for evaluation, and the test temperature. Properly hot-cold-worked or as-forged materials, except for Inconel-X, have the best properties up to 1200 F. Differences in rupture strength resulting from differences in processing or fabrication decrease with increase of testing time and with increase in testing temperature. Low ductility in the rupture test accompanies severe hot-cold work, particularly when a solution treatment is used prior to hot-cold work. At 1350 F., solution-treated-and-aged materials have properties equal to or better than the hot-cold-worked and forged materials, and at 1500 F. are definitely superior. Considerable variability in properties is sometimes obtained in as-forged materials, depending on forging conditions, so they may show properties ranging from those usually considered representative of hot-cold-worked materials to those more like solution-treated and aged materials. There were some indications that aging of solution-treated material at 1350 F. to 1400 F. produces higher strengths than aging at 1500 F.

There are no consistent relationships between tensile properties and rupture, creep, or stress-time for total deformation characteristics. The rupture strengths do not follow the trend of the tensile properties and in particular do not follow the trend of the yield strengths at 1350 and 1500 F. The creep strengths follow the trend of the rupture strengths, but comparison of the creep strengths with the rupture strengths indicates that creep strengths are not a reliable, and are sometimes a dangerous, basis of extrapolation for determination of long-time load-carrying ability for hot-cold-worked materials at 1200 F. or for any of the disk materials and treatments considered in this investigation at 1350 and 1500 F. The strengths for amounts of total deformation up to 1 per cent tend to follow the same trends as the rupture strengths, and neither the strengths for total deformation, creep, or rupture appear to bear any relationship to the elongations measured either in the tensile or the rupture tests. In other words, elongations in the tensile tests, or elongations in the rupture tests for a given rupture time bear no relationship to the tensile load-carrying ability of the materials tested in this investigation within the limits of permissible deformation for practical operation in a gas turbine.

At 1200 F. the variations in properties of the various alloys are such that tensile load-carrying ability would be much the same for all the alloys, except S-816 and Inconel-X, with proper control of processing and treatment. The less highly alloyed materials, particularly when hot-cold worked, have comparatively low load-carrying ability at 1350 and 1500 F. The relative strengths at 1350 and 1500 F. of low-carbon N-155, S-590, and S-816 disks are to a considerable extent dependent on heat treatment. Alloy S-816, and to a greater extent Inconel-X, have greater inherent load-carrying ability at 1200 F. The degree of superiority decreases at 1350 F. At 1500 F., the differences in strength between the more highly alloyed materials are relatively minor and dependent on treatment. Inconel-X shows entirely different behavior from the other alloys in that the transition point at which "third-stage" creep begins occurs at short testing times and at low total deformations.

In this paper, complete comparisons between the properties of experimental bar stock and large forgings have not been included. The authors are reasonably certain, however, that the properties of bar stock are reproduced at the rims of such forgings to the degree to which the processing and heat-treatment conditions used on bar stock are reproduced in the large forgings. The strengths of the Timken, CSA, and EME alloy disks are below those which have been attained experimentally on bar stock. The properties of the Timken alloy disks represent those which have resulted from the development of a feasible commercial production process. There is every reason to expect that commercial processing and treatment of large disks of CSA and EME alloys could be developed to give improved properties, although the attainment of the ultimate properties produced in experimental bar stock may not be commercially feasible. The 19-9DL data are a good example of the variation in properties which results from different processing and treatment schedules.

In general, the properties of the heat-treated higher-alloyed disks approximated reasonably well the properties which have been obtained from similarly treated bar stock. The as-forged disks, however, may or may not have had the same properties as bar stock depending on the differences in forging procedures used in their preparation.

REFERENCES

(1) J. W. Freeman, E. E. Reynolds, and A. E. White, "A Metallurgical Investigation of Five Contour-Forged Gas-Turbine Discs of Timken Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1531* (1947).

(2) J. W. Freeman, E. E. Reynolds, and A. E. White, "A Metallurgical Investigations of a Large Forged Disc of CSA (234-A-5)," Nat. Adv. Com. Aeronautics, *ARR No. 5H17* (1945).

(3) E. E. Reynolds, J. W. Freeman, and A. E. White, "A Metalurgical Investigation of Two Large Discs of CSA Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1533* (1947).

(4) E. E. Reynolds, J. W. Freeman, and A. E. White, "A Metallurgical Investigation of a Contour-Forged Disc of EME Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1534* (1947).

(5) J. W. Freeman, E. E. Reynolds, and A. E. White, "A Metallurgical Investigation of a Large Forged Disc of 19-9DL Alloy," Nat. Adv. Com. Aeronautics, *ACR No. 5C10* (1945).

(6) J. W. Freeman, E. E. Reynolds, and A. E. White, "A Metallurgical Investigation of Two Contour-Forged Gas-Turbine Discs of 19-9DL Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1532* (1947).

(7) E. E. Reynolds, J. W. Freeman, and A. E. White, "A Metallurgical Investigation of Two Turbosupercharger Discs of 19-9DL Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1535* (1947).

(8) J. W. Freeman and H. C. Cross, "A Metallurgical Investigation of a Large Forged Disc of Low-Carbon N-155 Alloy," Nat. Adv. Com. Aeronautics, *ARR No. 5K20* (1945); *OSRD No. 6427*, Serial No. M-617, August 4, 1945.

(9) Howard C. Cross and J. W. Freeman, "A Metallurgical Investigation of Large Forged Discs of Low-Carbon N-155 Alloy," Nat. Adv. Com. Aeronautics, *TN No. 1230* (1947). Office of Naval Research Report, Navy Department, December, 1946.

(10) J. W. Freeman and Howard C. Cross, "A Metallurgical Investigation of Two Large Forged Discs of S-590 Alloy," Nat. Adv. Com. Aeronautics, *TN No.* 1760, 1948. Office of Naval Research Report, Navy Department, 1948.

(11) Howard C. Cross and J. W. Freeman, "A Metallurgical Investigation of a Large Forged Disc of S-816 Alloy," Nat. Adv. Com. Aeronautics, *TN No.* 1765, 1948. Office of Naval Research Report, Navy Department, 1948.

(12) J. W. Freeman and Howard C. Cross, "A Metallurgical Investigation of a Large Forged Disc of Inconel-X Alloy," Nat. Adv. Com. Aeronautics, *TN No.* 1770, 1948. Office of Naval Research Report, Navy Department, 1948.

(13) Howard C. Cross and Ward F. Simmons, "Heat-Resisting Metals for Gas Turbine Parts," Symposium on Materials for Gas Turbines, Am. Soc. Testing Mats., Technical Publication No. 68, June, 1946.

(14) J. W. Freeman, E. E. Reynolds, and A. E. White, "High Temperature Alloys Developed for Aircraft Turbosuperchargers and Gas Turbines," Symposium on Materials for Gas Turbines, Am. Soc. Testing Mats., Technical Publication No. 68, June, 1946.

DISCUSSION

MR. A. R. BOBROWSKY[1] (*presented in written form*).—The authors have presented a paper which contains the most comprehensive data obtained to date on the properties of high-temperature alloys in gas turbine disks. Their results have produced information sufficiently complete to make possible prediction of behavior of disks of new design. The discussion of results goes beyond this by supplying for the first time general principles on which mechanical and thermal processing procedures may be based. Hot-cold working, for example, is now a known tool which the metallurgist can utilize with a fair degree of confidence.

A major problem still remaining for solution is the correlation of the elevated temperature properties of these materials with their performance in service. True, given the temperature and stress distribution in a disk, the time for maximum allowable creep can be obtained by existing analytical methods from the data presented. But such factors as ductility are meaningless unless the effect of ductility on burst speed of a given disk is known. Research is proceeding at present at a number of laboratories on these aspects of disk behavior, and it is hoped that the results thereof will round out the present data.

With respect to the existing question as to whether the log-log or the semi-log plot of stress rupture data is the more fitting, it may be noted that a great many of the data yield straight lines on semi-log plots, some yield straight lines on log-log plots, and some fit neither criterion. I wonder whether the authors would care to comment on the rôle played by structural instability in affecting the shape of these graphs.

MR. LEO SCHAPIRO.[2]—I should like to ask a few questions about the tests. In the basic data shown in the figures, does each point represent a single test per disk?

I should also appreciate knowing how the specimens are taken with respect to the thickness of the disk since the thicknesses were not all similar.

MR. O. E. HARDER.[3]—Obviously this work gives a great deal of new information. We are finding the properties of the materials with different types of processing. There is, however, a need—and I am sure the work is being done on this—of correlation of microstructures, etc., with the properties obtained. In other words, how is the variation in the properties brought about as a result of these heat treatments?

There is another phase that may need to be in the picture ultimately, and that is the correlation of the resistance of the materials to thermal fatigue or thermal shock, with processing and microstructures. It has been shown in some other work, not a part of the research being reported upon here, that there is a pronounced variation in the resistance of materials to failure by thermal fatigue or thermal shock, and their resistance to deformation in thermal cycles. For a given material or given type of alloy, there is a trend, perhaps, for the resistance to thermal fatigue to decrease as the load-carrying capacity or the creep resistance is increased by changes in composition or methods of processing. On the other hand, if an attempt is made to avoid failure by thermal fatigue, then there is a trend to encounter failure by deformation. Ultimately there will need to be optimum combinations of resistance to thermal fatigue and of resistance to deformation. In some applications thermal fatigue will be dominant, in others resistance to deformation will be more important. That type of work may need to get into this research program.

---

[1] Lewis Flight Propulsion Lab., National Advisory Committee for Aeronautics, Cleveland, Ohio.

[2] Chief Metallurgist, Materials Lab., Douglas Aircraft Co., Inc., Santa Monica, Calif.

[3] Assistant Director, Battelle Memorial Institute, Columbus, Ohio.

MR. J. W. FREEMAN (*author's closure*).—The authors are grateful to Messrs. Bobrowsky and Harder for their comments. Mr. Bobrowsky introduces the question of burst speeds and ductility. This paper gave no consideration to these questions other than to supply basic physical properties which might be used to correlate with performance of disks. Major emphasis was placed on high-temperature properties of rim materials rather than on disk centers which normally control burst characteristics in the usual overspeed test. The effect of time at temperature under service conditions on burst characteristics remains to be demonstrated.

According to some theories of plastic flow, the stress-rupture time data should give a hyperbolic sine curve. This curve approximates a straight line when stress is plotted against the logarithm of time for fracture except when the stress approaches zero. These theories do not, however, give consideration to changes in the test bars due to structural alteration or corrosion during testing. These factors probably account for the variable types of curves when the data are plotted to log-log and to semi-log coordinates. In practice the log-log curves have been found most useful. It should be mentioned that the semi-log plots used for the design curves were not necessarily based on theoretical grounds but were used because that coordinate system makes a convenient graph of the data.

The comments of Mr. Harder point out some of the problems of the metallurgy of gas turbine disks and emphasizes the need for more information on the reasons for the effects, as well as knowing what metallurgical effects result from specific treatments on specific disks. It is to be hoped that theoretical studies will be made to explain the results.

Mr. Schapiro asked about the number of tests. At every temperature there were at least five or six rupture tests taken and at least two long-time creep tests. The points given on most of the diagrams shown were taken from the so-called design data curves. There may have been some variation of the properties of individual specimens which may have influenced the results some. But in general the results were fairly good average values, at least for the disks tested. Exact records of where every specimen came from in each disk are available, although too lengthy for inclusion in this paper.

FACTORS OF IMPORTANCE IN THE ATMOSPHERIC CORROSION TESTING OF LOW-ALLOY STEELS*

BY H. R. COPSON[1]

SYNOPSIS

Some complications in the atmospheric corrosion testing of low-alloy steels are brought out. Data are presented which show that it is unsafe to draw general conclusions from tests of limited character. The results obtained depend on the location (whether industrial, marine, or rural), on the duration of the tests (the benefit of alloying increases with time), on the manner of exposure (particularly whether sheltered or boldly exposed), on the method of estimating corrosion (whether weight loss, pit depth, or time to disintegration), and on the weather (particularly as regards wetness and pollution). Under all conditions alloying seems of value, but the magnitude of the benefit and the relative merit of different steels and alloying elements varies. For example, a few weight loss *versus* time curves have been found to cross. In a thin steel, pit depths become more important than weight losses. Likewise for short exposures the benefit to be obtained by complex alloying may not be worth the additional cost. These effects are illustrated chiefly by means of data obtained through five years of exposure of 71 low-alloy steels at a marine location at Block Island, R. I., and an industrial location at Bayonne, N. J.

A frequent method of corrosion testing is exposure to the atmosphere. Study of published reports shows that many factors may influence results and conclusions. It is significant that, in spite of widespread interest, the A.S.T.M. has not developed a recommended standard method for conducting atmospheric corrosion tests, although the subject has received attention **(1.)**[2]

In the present paper, factors of importance in atmospheric corrosion are discussed. The discussion pertains particularly to low-alloy steels; other materials may behave differently. Most of the data have been drawn from exposure tests started by this laboratory in 1941 **(2)**. The data are supplemented to some extent by results drawn from other tests being conducted by this laboratory **(3)**, and from tests at Kure Beach, N. C. **(4)**.

Some of the factors which need to be considered are indicated by the following questions:

1. Where will the tests be made? It makes a difference whether the atmosphere is industrial, marine, rural, or some combination or modification of these.

2. How long should the tests be continued? Different conclusions may be drawn depending upon whether the tests are continued for one month or for several years.

3. How will the specimens be mounted? It makes a difference whether the specimens are sheltered or boldly exposed, in-

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Research Chemist, The International Nickel Co., Inc., Research Laboratory, Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 606.

TABLE I.—ANALYSES AND THICKNESSES OF STEELS.

Steel	Group	Description	Thickness, in.[a]	Analysis, per cent									
				C	Mn	Si	S	P	Ni	Cu	Cr	Mo	V
No. 44	I	High purity iron, plus copper	0.078	0.016	0.016	0.002	0.03	0.005	0.05	0.014			
No. 45			0.071	0.020	0.020	0.003	0.03	0.006	0.05	0.020			
No. 63			0.079	0.020	0.023	0.002	0.03	0.005	0.05	0.053			
No. 54			0.049	0.02	0.07	0.01	0.03	0.003	0.18	0.10			
No. 55			0.047	0.01	0.06	0.01	0.03	0.003	0.13	0.27			
No. 10	II	Low phosphorus steel, plus copper	0.050	0.041	0.22	0.002	0.04	0.010	0.05	0.02	0.01		
No. 31			0.031	0.09	0.35	0.05	0.02	0.009	0.05	0.041			
No. 5			0.029	0.043	0.39	0.007	0.02	0.007	0.01	0.05	0.07		
No. 57			0.049	0.06	0.26	0.01	0.03	0.007	0.06	0.19		0.01	
No. 6			0.030	0.046	0.40	0.005	0.02	0.007	0.000	0.21	0.07		
No. 41			0.076	0.08	0.34	0.01	0.02	0.008	0.05	0.22			
No. 43			0.080	0.06	0.32	0.01	0.02	0.010	0.05	0.24			
No. 64			0.083	0.026	0.24	0.004	0.04	0.009	0.05	0.265			
No. 40			0.064	0.03	0.17	0.01	0.03	0.006	0.05	0.27			
No. 58			0.061	0.05	0.31	0.01	0.02	0.008	0.05	0.27			
No. 42			0.076	0.08	0.42	0.01	0.02	0.010	0.05	0.30			
No. 56			0.049	0.04	0.31	0.01	0.02	0.004	0.05	0.30		0.01	
No. 7			0.028	0.038	0.40	0.004	0.02	0.008	0.004	0.50	0.07		
No. 1	III	High phosphorus steel, plus copper	0.057	0.085	0.43	0.005	0.05	0.103	0.002	0.01	0.02		
No. 11			0.033	0.040	0.49	0.003	0.04	0.074	0.01	0.05	0.10	0.003	
No. 2			0.063	0.095	0.41	0.007	0.06	0.106	0.003	0.20	0.03		
No. 12			0.033	0.026	0.47	0.003	0.06	0.066	0.03	0.27	0.03	0.003	
No. 16			0.034	0.09	0.43	0.005	0.03	0.058	0.24	0.36	0.06		
No. 3			0.058	0.095	0.41	0.007	0.05	0.104	0.002	0.51	0.02		
No. 18	IV	High manganese and silicon steels, plus copper	0.032	0.33	0.78	0.31	0.04	0.043	0.03	0.03	0.06		
No. 19			0.035	0.16	1.32	0.24	0.03	0.018	0.03	0.03	0.12		0.093
No. 17			0.029	0.25	1.35	0.18	0.03	0.022	0.01	0.28	0.03		
No. 66			0.077	0.17	0.67	0.23	0.03	0.012	0.05	0.29	0.14		
No. 53			0.056	0.10	0.69	0.26	0.04	0.072	0.06	0.43			
No. 67	V	Copper steel, plus chromium and silicon	0.062	0.07	1.52	0.75	0.02	0.017	0.08	0.10	0.40		
No. 68			0.060	0.22	1.13	0.65	0.02	0.06	0.04	0.12	0.50		
No. 154			0.049	0.17	0.79	0.89		0.017	0.10	0.22	0.60		
No. 97			0.038	0.13	0.66	0.79	0.02	0.029	0.11	0.22	0.56		
No. 13			0.035	0.072	0.27	0.83	0.02	0.140	0.03	0.46	1.19		
No. 118	VI	Copper steel, plus molybdenum	0.075	0.04	0.15	0.01	0.03	0.009	0.05	0.42		0.07	
No. 120			0.077	0.04	0.12	0.01	0.02	0.010	0.05	0.47		0.07	
No. 38			0.059	0.21	1.04	0.23	0.03	0.017	0.036	0.62	0.025	0.35	0.006
No. 121			0.073	0.06	0.096	0.01	0.03	0.069	0.14	0.54		0.13	
No. 48			0.072	0.17	0.89	0.05	0.03	0.075	0.16	0.47		0.28	
No. 32	VII	Nickel steel	0.032	0.11	0.69	0.14	0.02	0.011	0.20	0.038			
No. 27			0.027	0.16	0.56	0.23	0.03	0.013	0.54	0.12	0.05		
No. 33			0.032	0.14	0.37	0.07	0.02	0.008	0.55	0.03			
No. 34			0.032	0.14	0.36	0.37	0.02	0.008	1.27	0.028			
No. 28			0.028	0.14	0.47	0.23	0.02	0.014	1.47	0.03	0.05		
No. 147			0.030	0.06	0.14	0.14	0.03	0.013	1.96	0.03			
No. 46			0.029	0.16	0.57	0.020	0.02	0.015	2.20	0.24			
No. 35			0.033	0.14	0.28	0.08	0.02	0.008	2.55	0.03			
No. 20			0.030	0.19	0.53	0.009	0.02	0.016	3.23	0.07			
No. 62			0.046	0.17	0.58	0.26	0.01	0.007	4.98	0.09			
No. 36			0.035	0.13	0.23	0.07	0.01	0.007	4.99	0.03	0.05		
No. 30	VIII	Nickel steel plus chromium	0.030	0.13	0.45	0.23	0.03	0.017	1.18	0.04	0.65	0.01	
No. 29	IX	Nickel steel plus molybdenum	0.030	0.16	0.53	0.25	0.01	0.013	1.84	0.03	0.09	0.24	
No. 51	X	Nickel steel plus chromium and molybdenum	0.036	0.10	0.59	0.49	0.01	0.013	1.02	0.09	1.01	0.21	
No. 52			0.035	0.08	0.57	0.33	0.01	0.015	1.34	0.19	0.74	0.25	
No. 37	XI	Nickel copper steel	0.063	0.09	0.66	0.044	0.02	0.106	0.64	1.12			
No. 60			0.042	0.12	0.57	0.17	0.02	0.01	1.00	1.05			
No. 148			0.030	0.05	0.17	0.13	0.03	0.014	1.05	0.89			
No. 50			0.028	0.09	0.48	1.00	0.03	0.055	1.14	1.06			
No. 39			0.067	0.11	0.43	0.18	0.02	0.012	1.52	1.09			
No. 149			0.031	0.05	0.32	0.20	0.07	0.043	1.58	0.86			
No. 150			0.032	0.05	0.13	0.13	0.03	0.012	1.77	0.93			
No. 153			0.029	0.06	0.27	0.20	0.01	0.013	2.78	1.83			
No. 152			0.032	0.06	0.14	0.41	0.03	0.015	2.82	1.30			
No. 151			0.032	0.08	0.20	0.21	0.01	0.015	3.12	1.14			

TABLE I.—ANALYSES AND THICKNESSES OF STEELS. (*Continued*)

Steel	Group	Description	Thickness, in.[a]	Analysis, per cent									
				C	Mn	Si	S	P	Ni	Cu	Cr	Mo	V
No. 49	XII	Nickel copper steel plus chromium	0.074	0.09	0.33	0.01	0.03	0.066	0.4	0.22	0.19		
No. 16-1			0.032	0.044	0.20	0.003	0.02	0.010	0.49	0.54	0.46		
No. 59			0.065	0.11	0.65	0.13	0.02	0.086	0.29	0.57	0.66		
No. 47			0.031	0.11	0.75	0.23	0.04	0.020	0.65	0.53	0.74		
No. 14			0.052	0.08	0.37	0.29	0.03	0.089	0.47	0.39	0.75		
No. 119	XIII	Nickel copper steel plus molybdenum	0.079	0.03	0.16	0.01	0.03	0.009	0.29	0.53		0.08	
No. 65			0.079	0.13	0.45	0.066	0.02	0.073	0.73	0.573		0.087	

[a] Generally within ±0.002 in.

clined or vertical. It may make a difference whether they face south or east or some other direction. Specimen size, shape, and thickness must be considered, and also the elevation above ground level.

4. How will the results be evaluated? Weight losses may not lead to the same conclusions as pit depth measurements or time to disintegration.

5. Will the weather affect the results? It may make a difference what season of the year the specimens are exposed. The weather immediately following exposure may affect subsequent behavior and influence the reproducibility of the results. Long-time trends or changes in weather conditions will affect reproducibility.

6. What records should be kept of at-

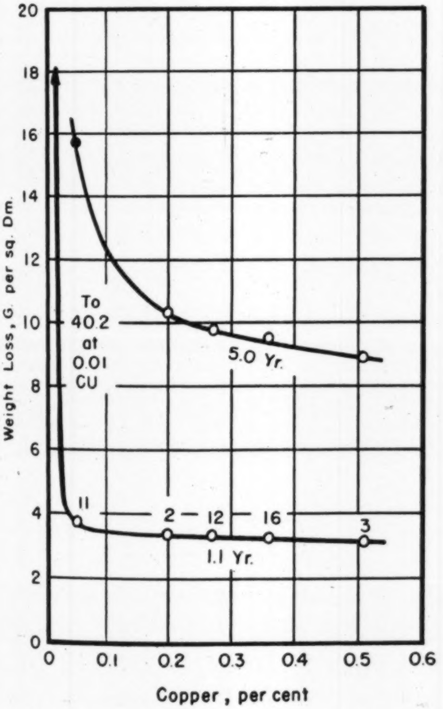


FIG. 1.—Weight Loss *versus* Copper Content of Group III, High Phosphorous Steels at Block Island.

Blacked in point indicates perforated specimen.

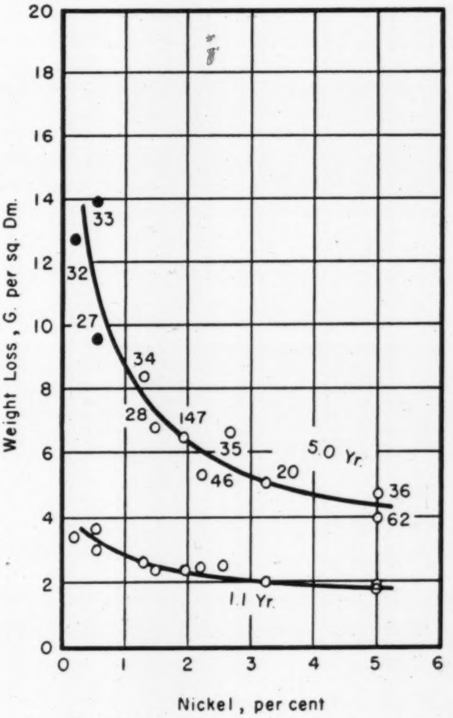


FIG. 2.—Weight Loss *versus* Nickel Content of Group VII Nickel Steels at Block Island.

Blacked in points indicate perforated specimens.

mospheric conditions? With sufficient data it should be possible to predict weight losses and corrosion behavior in an untried atmosphere, but at present any detailed prediction is dangerous. Subcommittee VII of A.S.T.M. Committee B-3, has as its object the correlation of weather with atmospheric corrosion.

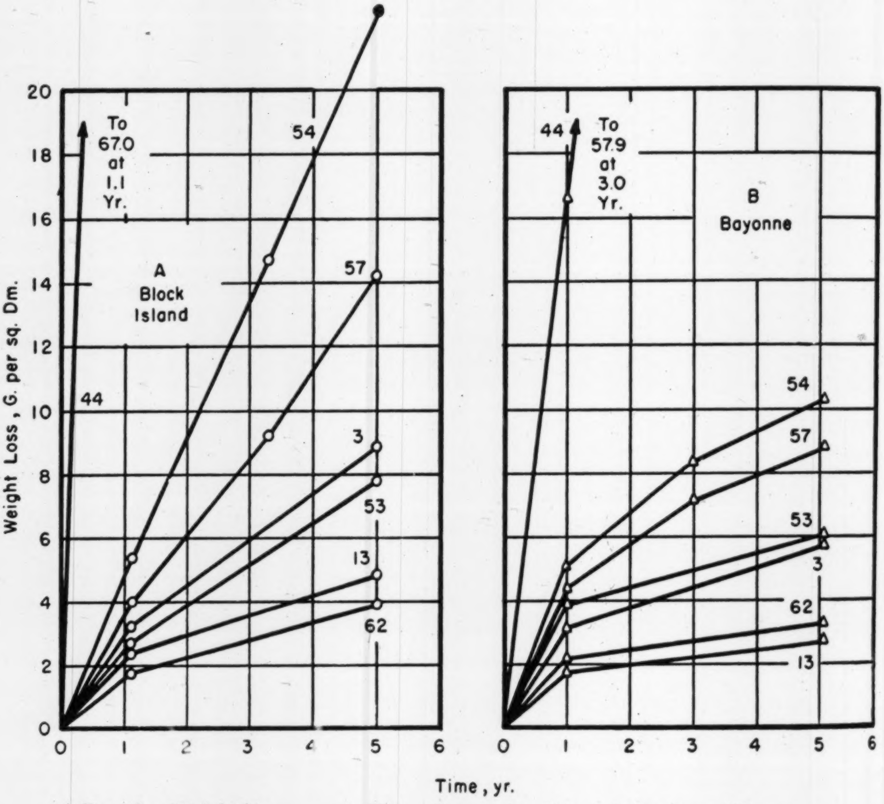


FIG. 3.—Weight Loss *versus* Time Curves for Selected Steels and Irons.
Blacked in point indicates perforated specimen.

ATMOSPHERIC TESTS STARTED IN 1941

Five sets of 71 low-alloy structural steels were exposed to the atmosphere in May, 1941, at two locations (2).

One of the locations was in the yard of this laboratory at Bayonne, N. J., not far from a center of oil refining activity. This is considered an industrial atmosphere. The other location was at Block Island, R. I. The test site is about 15 miles from the mainland, and is remote from manufacturing centers. This is considered a marine location. The analyses and thicknesses of the steels are given in Table I.

The 4- by 6-in. pickled specimens of the main exposures were exposed at an angle of 30 deg. from the horizontal and faced south. Other details have already been described (2).

EFFECT OF ALLOYING ELEMENTS

It is well known that certain alloying elements improve the corrosion behavior of steel in the atmosphere (5, 6). Some new relationships are established below but first some of the better known effects will be described.

Figure 1 shows the effect of copper content on the weight loss of the group III, high phosphorus steels at Block Island, both at 1 and 5 yr. A little copper was very beneficial, but the effect of additional copper was less marked. The phosphorus content of these steels was helpful.

Figure 2 shows the effect of nickel content on the weight loss of the group VII steels at Block Island, both at 1 and 5 yr. A little nickel was not as effective as a little copper, but at higher concentrations nickel was more effective, the beneficial effect of nickel continuing to increase with nickel content. All these nickel steels contained at least about 0.03 per cent copper.

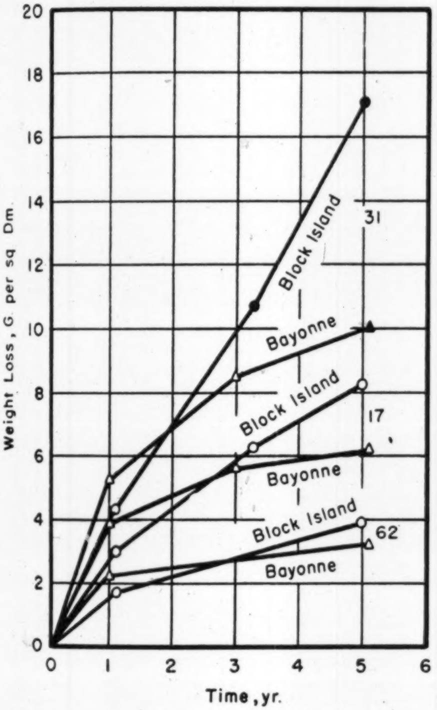


FIG. 4.—Weight Loss *versus* Time Curves for Three Steels.

Blacked in points indicate perforated specimens.

Chromium also has a beneficial effect and some weight loss composition curves have been published by Binder and Brown (7). When several elements are added at the same time results are impossible to predict (8). An element such as silicon which does not have much effect by itself may be decidedly beneficial when added along with chromium, copper, and phosphorus. An optimum amount of copper in nickel steels improves their behavior, in some atmospheres at least, over that of steels with the same total of nickel or copper alone (3). On the other hand the beneficial effects of most combinations are less than additive. Some elements which may be effective by themselves do not show up well when added in combination with other elements.

Figure 3 shows some weight loss-time curves for a few selected steels at Bayonne and Block Island. Additional curves of this type appear in the literature (9). For the better steels, corrosion rate decreases rapidly with time, showing that the rust coating is protective. This complicates a comparison of the relative merit of two steels. Thus at Bayonne, steel No. 62 was corroding more than half as fast as steel No. 3, but it took five times as long to reach the weight loss acquired by steel No. 3 in one year.

TABLE II.—EFFECT OF ATMOSPHERE ON RELATIVE MERIT OF SOME STEELS.

Group	Steel	Group Number in Table I	Five Year Weight Loss, g. per sq. dm.	
			Bayonne[a]	Block Island[b]
A	No. 13	V	2.64	4.83
	No. 153	XI	2.94	4.52
	No. 51	X	3.17	4.43
	No. 62	VII	3.24	3.93
B	No. 148	XI	3.89	5.89
	No. 30	VIII	5.10	5.53
	No. 46	VII	5.30	5.37

[a] Note increasing values of figures in column.
[b] Note decreasing values of figures in column.

EFFECT OF ATMOSPHERIC POLLUTION

One of the most important considerations in atmospheric corrosion testing is the nature of the local atmosphere, particularly as to humidity and purity. This was emphasized by the laboratory work of Vernon (10). Vernon found corrosion to be negligible below a humidity of 50 to 60 per cent, and even at higher humidities corrosion was not severe in pure air. However, in the presence of a little sulfur dioxide corrosion was rapid once a critical humidity of about 80 per cent was exceeded. In the presence of certain solids such as ammonium sulfate and charcoal, the effect of sulfur dioxide was even more pronounced. Atmospheric pollution with these substances is common in industrial centers.

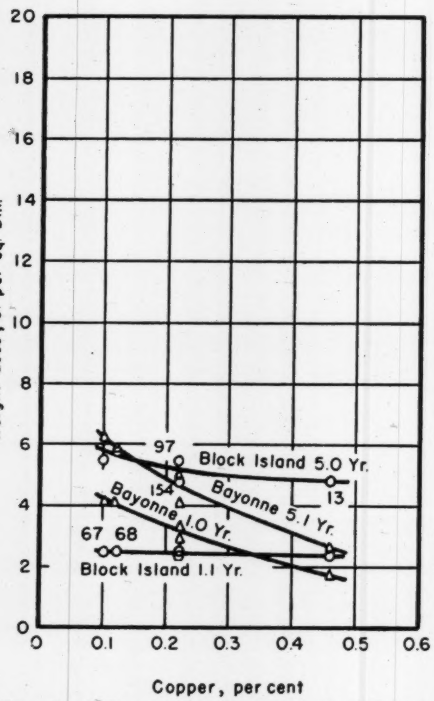


FIG. 5.—Effect of Copper in Steels of Group V with High Phosphorous, Chromium, and Silicon.

The Corrosion Committee of the British Iron and Steel Institute has made exposure tests at several locations throughout the world (11). They found the relative corrodability of the different atmospheres to vary from about 1 to 100. They also found results to vary from year to year with some years 2 to 3 times as corrosive as others. In accord with Vernon they found corrosion to be slow in dry and rural locations, and fast in industrial centers.

On the basis of their one year results on a pure ingot iron, it would be concluded that, in general, industrial atmospheres were more corrosive than marine. On the contrary it is suspected that had the tests been of long enough duration, and particularly if a low-alloy steel had been used, then very likely the reverse conclusion would have been drawn (12). This is illustrated for three steels in Fig. 4. At one year the weight losses for each steel were higher in the industrial Bayonne atmosphere than in the marine Block Island atmosphere. By three years the weight loss-time curves crossed, and by five years the weight losses for each steel were markedly higher in the marine atmosphere than in the industrial atmos-

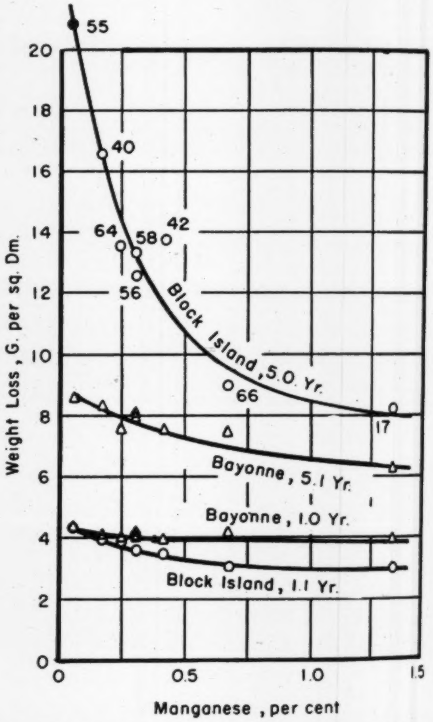


FIG. 6.—Effect of Manganese in Low Phosphorous 0.3 per cent Copper Steels.

Blacked in point indicates perforated specimen.

phere. This effect can also be seen in Fig. 3 and appears to be quite general. A possible explanation is offered below.

In Fig. 4 the three steels shown are rated in the same order at the two locations. This is only because they were chosen that way to keep the graph simple. In Fig. 3, steels 13 and 62, and 3 and 53 would be rated differently in the two atmospheres, and it is a simple matter to select steels which would be rated in reverse order. This has been done for two groups in Table II. In each group the straight nickel steel would be rated best in the marine Block Island atmosphere, whereas in the industrial Bayonne atmosphere additional alloying with chromium and copper gave improved results. To some extent the variations within the groups may be minor because all these steels have good resistance to atmsopheric corrosion. This is shown by comparing the behavior of a fast corroding iron, such as No. 44 in Fig. 3.

TABLE III.—EFFECT OF TIME IN BRINGING OUT DIFFERENCES.

Steel	Group No. in Table I	Weight Losses at Bayonne, g. per sq. dm.		
		1.0 yr.	5.1 yr.	Increase
No. 68	V	4.09	5.91	1.82
No. 53	IV	3.96	6.05	2.09
No. 67	V	4.10	6.20	2.10
No. 7	II	4.03	6.88	2.85
No. 66	IV	4.13	7.46	3.33
No. 118	VI	4.02	7.72	3.70
No. 43	II	4.07	7.98	3.91
No. 41	II	4.07	8.03	3.96
No. 58	II	4.00	8.05	4.05
No. 40	II	4.08	8.30	4.22

That copper additions are more beneficial at Bayonne than at Block Island is also shown by Fig. 5. For the steels of group V, copper contents above about 0.1 per cent were without much effect on the weight loss at Block Island. On the contrary, at Bayonne weight loss decreased steadily with copper content up to 0.5 per cent copper at least.

It has been stated in the literature that manganese was without much effect on atmospheric corrosion of steel **(5, 11)**. Figure 6 shows that such broad inferences are dangerous. In the marine Block Island atmosphere, manganese is decidedly beneficial in low-phosphorus, 0.3 per cent copper steels, and the amount of benefit seems to be increasing with time. In the industrial Bayonne atmosphere, manganese had no detectable effect on the weight losses of these steels at one year, but by five years some benefit was showing.

EFFECT OF TIME

In discussing Fig. 4 it was pointed out that the relative corrosivity of marine and industrial atmospheres may change with time. In discussing Fig. 6 it was pointed out that the benefit derived from additions of manganese increased with time.

TABLE IV.—EFFECT OF TIME ON RELATIVE MERIT OF SOME STEELS.

Group	Steel	Group No. in Table I	Weight Losses, g. per sq. dm.				
			Bayonne			Block Island	
			1.0 yr.[a]	3.0 yr.[b]	5.1 yr.[b]	1.1 yr.[a]	5.0 yr.[b]
A	No. 30	VIII	3.70	4.42	5.10		
	No. 60	XI	3.48	4.49	5.32		
	No. 28	VII	3.29	4.56	5.82		
B	No. 13	V				2.40	4.83
	No. 30	VIII				2.30	5.53
	No. 29	IX				2.07	5.66

[a] Note decreasing values of figures in column.
[b] Note increasing values of figures in column.

This latter observation appears to be quite general for all alloy additions. When first exposed all steels corrode at about the same rate. This has been observed many times for short exposures lasting a month or so **(2, 13, 14, 15)**. As time goes on weight loss *versus* time curves diverge, and the position of the alloy steels improves (Fig. 3).

Even after one year steels which differ greatly in ultimate merit may have practically identical weight losses. This is indicated in Table III for 10 steels at Bayonne. During the first year these

10 steels corroded at the same rate, but from the first to the fifth year the best steel of this group corroded less than half as fast as the worst. The better steels in this group tended to be higher in alloying elements, in this case chiefly in manganese, chromium, and phosphorus.

Much the same point is brought out in Fig. 1 for the steels of group III. After one year at Block Island it might be concluded that there was little benefit to be derived from more than 0.05 per cent copper. However, from one to five years steel No. 11 with this amount of copper corroded more than twice as fast as steel No. 3 with 0.5 per cent copper. At five years there was steady improvement with increasing copper content. The inflection point in the curve was less pronounced and had shifted toward higher copper content.

As might be expected from these changes in relative corrosion rates with time, a few weight loss-time curves cross. In Table IV, after one year at Bayonne, the best steel in group A was nickel steel No. 28. By three years both the nickel copper steel, No. 60, and the nickel chromium steel, No. 30 had surpassed the straight nickel steel, and by five years the trend was even more pronounced. Similarly at Block Island, the group B steels also show the delayed benefit of complex alloying with copper and chromium. Chromium seems to be particularly effective in flattening the weight loss-time curves.

For short exposures, complex alloying may not be worth the additional cost. Thus for short times the straight nickel steels are as good or better than steels with nickel plus copper, or nickel plus chromium.

THEORY

The reasons for all the effects of local atmosphere and of the duration of exposure are not completely understood, but some ideas are well established. Vernon (10) showed the importance of humidity and of atmospheric pollution with sulfur dioxide. Nevertheless, he was primarily concerned with high purity iron (see reference (16), however), and with the initial stages of corrosion. As has been shown, it takes time for the value of alloy additions to develop.

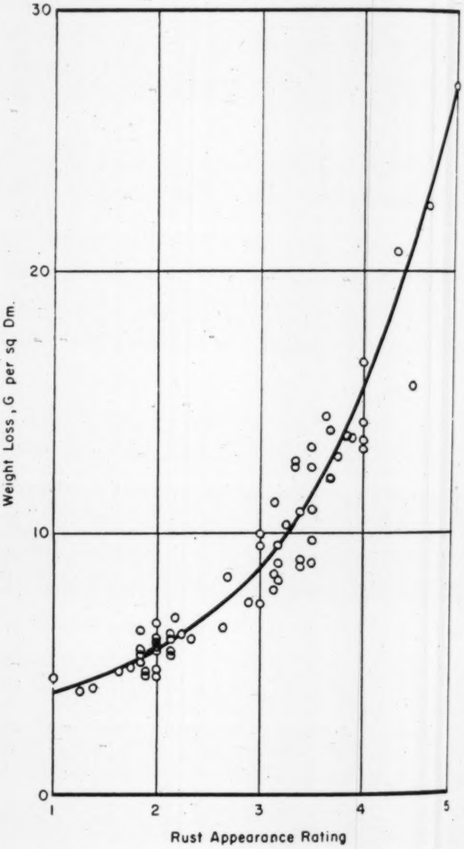


FIG. 7.—Rust Appearance Ratings *versus* Weight Losses of Block Specimens at Five Years.

The rust appearance ratings are the average of observations on three or four specimens; the average of all standard deviations is 0.19.

It has long been held that the rust coating on low-alloy steels was more protective than that on plain mild steel. The chief reason for this has been the appearance of the rust coating. Fast-corroding steels develop a thick, coarse, loose, light-colored rust, whereas good low-alloy steels develop a fine-textured, adherent, dark-colored rust. It is possible to rate the steels approximately just from their appearance.

This was done periodically, the specimens being arbitrarily rated 1 to 5, with 1 indicating the best appearing rust and 5 the worst. Fractional ratings were used when it seemed desirable. For the 3 to 5 sets of specimens on the racks at the inspection times, the average of all the standard deviations of the ratings was only about 0.2. The ratings at the different inspections were not directly comparable, primarily because the rust darkened with time.

Figure 7 shows the rust appearance ratings after five years at Block Island plotted against the subsequently determined weight losses. The smoothness of the curve confirms the ***approximate*** relationship between appearance of rust and corrosion rate. However, to a considerable extent the scatter in the plotted points is reproducible. Hence, basing conclusions or predictions on appearance alone can lead to appreciable error. For example, at Bayonne silicon in the steel darkened the rust without corresponding improvement in weight loss.

While appearance alone indicated that rust coatings differed greatly in their protective value, there has been no adequate explanation until recently as to how this difference might come about. Recently, analyses of rust coatings on copper and nickel steels showed that in industrial atmospheres the percentage of sulfate tied up in the rust increased as weight loss decreased (2). As a result it was postulated that copper and nickel in steel render sulfate corrosion products more insoluble by forming complex basic sulfates.

It then seemed evident that corrosion rate was controlled by the quality and quantity of water reaching the steel surface. Initially when the surface was bare all steels were exposed to the same environment and corroded at about the same fast rate. As corrosion products accumulated the amount of water reaching the steel surface became less. The solubility of the corrosion products then became important. Relatively insoluble products decreased the amount of soluble constituents in the water and thereby decreased corrosion. Relatively insoluble products would not wash away and thereby they decreased the porosity of the rust coating and decreased the quantity of water reaching the steel. As time goes on pores become plugged, the rust coating becomes more protective, and the corrosion rates of the low-alloy steels are relatively decreased.

TABLE V.—EFFECT OF NEARNESS TO OCEAN.
Specimens Exposed at Kure Beach, N. C., Jan. 19, 1944.

Steel	Group No. in Table I	Time, yr.	Weight Loss, g. per sq. dm. Average of 2 specimens	
			Rack 800 ft. from Ocean Facing South	Rack 80 ft. from Ocean Facing East
No. 1	III	0.35	3.29	15.50
		1.39	9.45	gone
No. 11	III	0.35	1.89	3.00
		1.39	4.33	gone
No. 6	II	0.35	1.94	3.26
		1.39	4.33	16.90[a]
No. 39	XI	0.35	1.74	2.26
		1.39	3.16	5.03

[a] Perforated.

Extensive analyses have not been made of rusts grown in marine atmospheres. However, a few analyses have shown surprisingly little chloride—less than 0.1 per cent—and surprisingly large amounts of sulfate—as much as 1 per cent or more (2). Hence the presence of sulfur gases in the atmosphere in small amount is probably important in marine atmospheres and in locations remote from manufacturing centers.

It must not be inferred that salt in the air is not important also. The importance of salt in marine air is shown by the weight loss-time curves in Fig. 4 and also by the effect of nearness to the ocean.

At Kure Beach, N. C., some identical specimens were exposed on racks located 80 ft. and 800 ft. from the ocean (4). Some typical results in Table V show that the specimens nearer the ocean corroded much faster than those further away, probably because they received more spray. Fast corroding steels were more affected than better low-alloy steels.

Nevertheless, deliberately adding salt to exposed specimens can lead to some curious results (8). Probably the amount of salt is not as important as the amount of iron chloride that may form in the rust coating. This suggests that a combination of sulfur dioxide and chlorides may be particularly damaging. An acid reaction due to sulfur dioxide would be expected to facilitate the formation of ferric chloride.

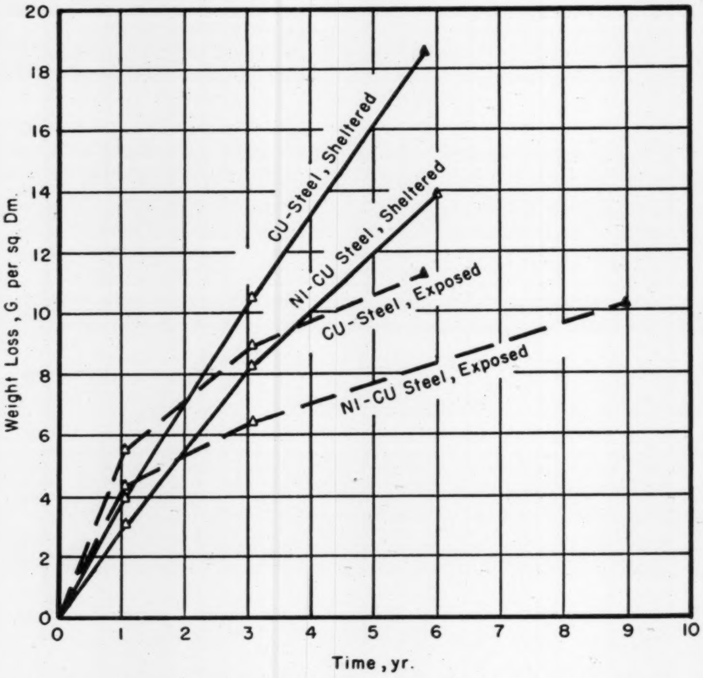


FIG. 8.—Effect of Shelter on Specimens Exposed Vertically at Bayonne.

	Composition, per cent						
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel
Cu-steel	0.03	0.30	0.008	0.053	0.01	0.29	...
Ni-Cu steel	0.05	0.32	0.045	0.064	0.20	0.86	1.58

Blacked in points indicate perforated specimens.

On the basis of this discussion it would be concluded that in industrial atmospheres the rust on low-alloy steels becomes more protective with time owing to the formation of insoluble basic sulfates, and the corrosion rates drop off to some very low values. In marine atmospheres the initial corrosion rate may not be as high as in industrial atmospheres, but the rust does not become as protective and corrosion continues at comparatively fast rates.

Further, it would be concluded that the washing away of soluble corrosion products has an important influence. If left on the specimen these soluble products increase subsequent corrosion. To a considerable extent, variation in results with the manner of exposure can be explained by the washing action of rain.

EFFECT OF EXPOSURE CONDITIONS

Shelter is one of the more important variables in exposure conditions. This has been discussed by Larrabee (17) and by Schikorr (18). Larrabee observed that on inclined metal specimens about 62 per cent of the total weight loss occurred on the groundward side of the specimens. On the other hand Schikorr stated that steel protected against rain rusted only one-half to three-fourths as much as steel exposed to rain. Some tests have been conducted by this laboratory on specimens mounted vertically, with one rack exposed boldly in the open and another rack mounted under a roofed shed open on all sides. This roofed shed has been described previously (19). Typical results from these tests are plotted in Fig. 8.

For a year or two weight losses were greater on boldly exposed specimens, probably because they were wet more often. However, the weight loss-time curves soon crossed and from two years on weight losses were greater on the sheltered specimens. This behavior is probably connected with the beneficial action of the wash of rain. This removes soluble corrosion products and permits insoluble products to plug pores. The rust accumulation on the sheltered specimens was powdery instead of scaly and not nearly as protective as on the exposed specimens.

Thickness measurements on the cleaned specimens at the end of these tests were instructive. On boldly exposed specimens the minimum lowering of the surface level as shown by flat micrometers was only about 0.001 in. On sheltered specimens it was greater than this measuring about 0.006 in. for the steels in Fig. 8. However, the maximum difference in level between adjacent points on the specimens also differed. On boldly exposed specimens the maximum difference in level was about 0.014 in., whereas on sheltered specimens it was only about half this or 0.007 in. Consequently it is evident that in these tests, in spite of the difference in weight loss, perforations might occur at about the same time under both exposure conditions.

On the inclined racks of the main exposure, small 4 by 6-in. specimens are not completely sheltered on the groundward surface. Measurements with flat micrometers on many cleaned specimens have indicated a minimum lowering of the surface level of less than 0.001 in. on both surfaces. On these inclined specimens, pits have consistently run about 0.002 in. deeper on the groundward surface than on the skyward.

In view of the above results it is not surprising that anything that provides shelter has some effect on the corrosion behavior. Taylerson (9) has summarized data on the effect of the angle of exposure. This has also been considered by Chaudron (20) and by Daeves, Mewes, and Schulz (21). Pilling and Wesley (3) in some tests designed to simulate actual conditions on a roof found disintegration to occur first in undrained rain troughs, next in sheltered areas, and last in exposed areas. Passano (22) noted that overlaps may be particularly damaging.

At Block Island strong winds have blown an occasional specimen off the racks. These specimens lay in long grass until the next inspection, when they were picked up and weight losses determined.

They always had somewhat greater weight loss than specimens remaining in position on the racks. This might be due to their being washed less thoroughly by rain and to their not drying out as fast.

Hudson (11) observed elevation to have opposite effects at different locations. He pointed out that in the immediate vicinity of the ground, wind velocity increased with height. Higher specimens might come in contact with more pollution, but they would also dry faster after dew or rain. Schikorr (23) cited an instance where due to the proximity of an exhaust stack, specimens 15 meters above ground corroded 30 to 50 per cent faster than specimens 1 meter above ground. In general, however, positional effects of elevation are small.

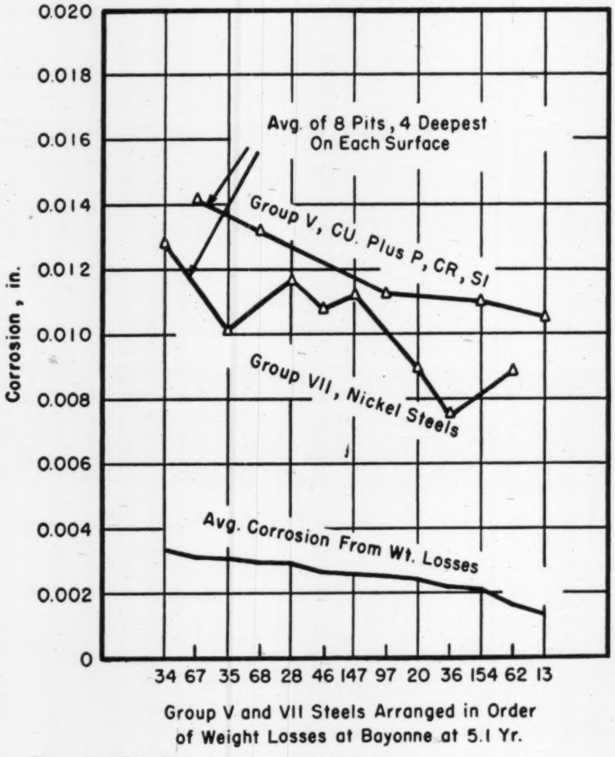


FIG. 9.—Pit Depths *versus* Weight Losses at Bayonne.

At Block Island another effect of exposure conditions was observed which was of considerable magnitude. This was the severe corrosion of certain edges of the specimens. The attack was always worse at the left or western edge, at the bottom edge nearest the ground, and under insulators at the right edge. This has been discussed previously (2), and was attributed to the predominant wind direction during rainfall. Prevailing wind from the east caused rain to wash soluble material on the specimens toward the western and groundward edges with the result that rapid perforation of some steels occurred at these areas. The more rapid attack was noticeable on both surfaces, although it was more local on the skyward than on the groundward surface. This is evidence that the groundward surface was not completely sheltered. The edge effects were most obvious on the fast corroding steels and barely detectable on the better alloy steels. Here is additional evidence that the alloy steels form relatively insoluble corrosion products.

To test the effect of exposure direction at this location a few selected specimens were exposed from June 5, 1945, to June 30, 1947, on a special small rack facing east at an angle of 30 deg. from the horizontal, along with controls on the large racks facing south. During this two-year period an average of 53 per cent of the total rain fell when the wind was from northerly directions.

In accord with this, corrosion was more severe at the edge opposite the predominant wind direction. This is considered confirmation of the importance of wash of rain at marine locations.

TABLE VI.—RATIO OF PIT DEPTH TO AVERAGE PENETRATION.

Weight Losses, g. per sq. dm.	Bayonne, 5.1 yr.				Block Island, 5.0 yr.			
	Number of Specimens Averaged	Average Weight Loss, g. per sq. dm.	Average Pit Depth, in.	Average of Pitting Factors[a]	Number of Specimens Averaged	Average Weight Loss, g. per sq. dm.	Average Pit Depth, in.	Average of Pitting Factors[a]
27.1					1	27.08	0.0319	2.34
19.7	1	19.70	0.0304	3.06				
11.3 to 16.6					10	13.82	0.0188	2.69
8.2 to 11.3	10	9.40	0.0160	3.37	12	9.34	0.0144	2.99
7.0 to 8.2	12	7.70	0.0138	3.54	10	6.79	0.0119	3.48
6.0 to 7.0	11	6.52	0.0126	3.85				
5.2 to 6.0	11	5.58	0.0111	3.95	11	5.63	0.0107	3.77
3.9 to 5.2	10	4.62	0.0104	4.48	10	4.54	0.0088	3.83
2.6 to 3.9	10	3.24	0.0094	5.71				

[a] Pitting Factor as here used is the average of 8 pits (the 4 deepest on each surface) divided by the weight loss, both in the same units.

EFFECT OF METHOD OF EVALUATING RESULTS

So far most of the emphasis has been on weight loss determinations with some mention of pit depths and visual observations. Since steel does not suffer intergranular or internal attack in the atmosphere, weight loss measurements are highly satisfactory. Nevertheless the chief interest may lie in the time to perforation, disintegration, or for a certain loss in strength.

Unfortunately perforations cannot be observed directly, any holes through the metal being completely obscured by the accumulation of rust. Some specimens which have appeared intact on the racks have been found on removing the rust to be little more than gauze or even to be completely corroded away. Thin specimens which were known to have perforated first at about one year still appeared intact on the racks after 13 years.

Owing to this behavior the observation of the time to disintegration or for holes to appear is not entirely satisfactory. To an appreciable extent the life of the rust is included in the life of the steel. In spite of this the time to disintegration is frequently the main criterion (**24**). Wide scatter is apt to occur in the observations.

Generally pit depths can be accurately measured on cleaned specimens (**25**). In the present work the 4 deepest pits on both surfaces of all specimens were measured after cleaning. This was accomplished by measuring the difference in level between adjacent points by means of a pointed metal probe attached to a dial gage. Since most of the high points were within 0.001 in. of the original surface, the measurements were considered to give the pit depths directly.

Careful examination of the results

showed that pit depths did not tell exactly the same story as weight losses. This is illustrated in Fig. 9. Here the steels of groups V and VII are arranged in the order of their 5-yr. weight losses at Bayonne. The high-nickel steels of group VII consistently have lower pit depths than the steels of group V. Steel No. 13 which would be rated best on the basis of weight loss is surpassed by several steels on the basis of pit depths. Probably this behavior is connected with the number of points of attack, with steels containing chromium tending to have fewer but deeper pits, and conversely steels containing nickel tending to have more but shallower pits. For a thin steel where perforation may be relatively fast, pit depths are more important than weight losses.

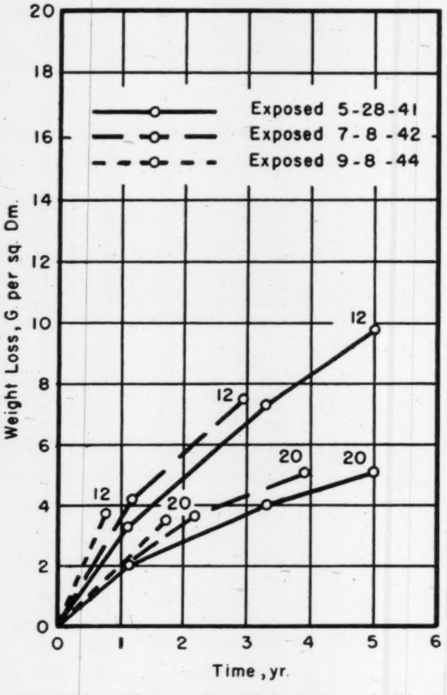


FIG. 10.—Change in Corrosivity of Block Island Atmosphere with Time.

Summarized data on the ratio of pit depth to average corrosion are given in Table VI. The pitting factors tend to increase as weight loss decreases. The pit depths for a given weight loss and the pitting factors tend to be somewhat smaller at Block Island than at Bayonne. Comparison with earlier results shows that the magnitude of the pitting factors was decreasing with time. These observations are related to the diameter and number of anodic areas.

Evidence of the unevenness of corrosion can also be obtained with the help of tension tests or measurements of electrical resistance (3). The latter method is nondestructive but requires specimens of special shape, long and narrow, which is apt to overemphasize any edge effects. The same defect resides in exposing tension specimens (26). For accurate weight losses it is desirable to have a large ratio of surface area to thickness. Regardless of the method of evaluation, the specimens should be thick enough to prevent early perforation and to allow time for the full benefit of alloying to appear.

TABLE VII.—WEATHER DATA AT BLOCK ISLAND, R. I.

		5-28-41 to 7-7-42 405 Days	7-7-42 to 8-31-43 419 Days	8-31-43 to 9-7-44 374 Days	9-7-44 to 6-5-45 271 Days	6-5-45 to 5-30-46 359 Days
Actual rainfall	inches...	38.55	44.52	31.21	35.27	37.63
	per cent of normal...	85.7	94.7	74.3	111.2	92.1
Rain from Northerly Semicircle per cent..		26.4	30.0	40.4	40.8	60.4
Days with more than 0.01 in rain	days....	125	142	113	103	126
	per cent.	30.9	33.8	30.3	38.2	35.1
Days with light fog	days....	157	219	197	141	218
	per cent.	38.8	52.2	52.8	52.2	60.7

EFFECT OF THE WEATHER

The corrosiveness of a given atmosphere is not constant but varies with the weather. Some years may be 2 to 3 times as corrosive as others (11). Schramm and Taylerson (27) observed marked differences in the corrosion of specimens put out at different seasons of the year and found a limited correlation with atmospheric pollution. Schikorr (28) established a relation between the rate of rusting and the average relative humidity. Wind direction on wet days may be important in determining the amount of pollution present (2). There seems to be no correlation between total rainfall and corrosion, probably because rain has a dual role. While moisture must be present for corrosion to occur, actual rainfall washes away soluble material.

In our tests, specimens of a few steels have been exposed periodically since the start of the main exposures in 1941. Figure 10 illustrates the effect of exposure date on weight loss-time curves at Block Island. At this location it appears that the corrosivity of the atmosphere has been increasing with time. Similar effects were less pronounced at Bayonne. In general, fast corroding steels seemed more affected than the better low-alloy steels.

In the weather data for Block Island summarized in Table VII, there is no indication that total rainfall, or the number of wet days, (days with more than 0.01 in. of rain) can account for the increase in corrosiveness with time. On the other hand, the yearly data from May 28, 1941, to June 30, 1946, the period covered by Fig. 10, indicate a steady increase in the amount of rain coming from northerly directions. This may mean an increasing amount of contamination blowing in from industrial cities to the north. The data also indicate an increase in the percentage of days with light fog. Thus the data seem to support the contention that pollution and humidity are important considerations.

EVALUATION OF ATMOSPHERIC VARIABLES

With sufficient data it should be possible to foretell corrosion behavior in an untried atmosphere. At present any detailed prediction is dangerous. Since weather cycles are varied and complex, and since conditions are continually changing, too much should not be expected. Some of the things that need to be known and studied further in order to increase the accuracy of predictions have already been mentioned.

Most important is atmospheric pollution. This requires detailed knowledge of the concentration and variation of corrosive gases such as sulfur dioxide and of solids such as chlorides, sulfates, and other active materials. It is obvious that the pollution will not be constant but will vary with industrial activity, with rainfall, humidity, wind direction, wind velocity, and atmospheric conditions.

Of nearly equal importance is the length of time conditions are propitious for rapid rusting. This is related to humidity, to fog, to the presence of hygroscopic solids, to the length of time wet, and is affected by temperature. Rainfall has to be considered because of its washing action both of the air and of the steel surface. Generally this is beneficial, but it may localize attack by concentrating corrosives at catchment areas.

Many of these factors are interrelated, and they all need to be studied further. They should be considered in connection with the composition of the steel. It is evident that any exact correlation of atmospheric conditions with corrosion behavior will require extensive data.

CONCLUSIONS

1. Weight loss-time curves for most steels in the Block Island atmosphere crossed the curves for the same steels exposed at Bayonne. At one year, the industrial atmosphere was the more corrosive, but by five years the reverse conclusion would be drawn.

2. Weight loss-time curves for certain steels in the same atmosphere crossed. Thus while a straight nickel steel seemed better at one year, replacing part of the nickel with chromium or copper produced better results by five years. Chromium seemed particularly effective in flattening weight loss-time curves. Nevertheless, for short exposures complex alloying would not be worth the additional cost.

3. Certain steels rated in reverse order at the two locations. Thus the additional alloying of nickel steels with copper or chromium seemed more effective at Bayonne than at Block Island.

4. The benefit derived from alloying increased with time. Even through one year, steels which differ greatly in ultimate merit may have practically identical weight losses.

5. Fractional percentages of manganese in copper steels were decidedly beneficial in marine atmospheres, and the benefit seemed to be increasing with time. The effect of manganese was much less pronounced at Bayonne.

6. In general, copper and chromium additions to steel were more effective at Bayonne that at Block Island, whereas manganese was more effective at Block Island. Nickel additions were effective at both locations.

7. Rust appearance gave an approximate indication of the corrosion. Fast corroding steels developed thick, coarse, loose, light-colored rust, whereas good low-alloy steels developed a fine textured, adherent, dark-colored rust.

8. Shelter had a deleterious effect on the protective character of rust. At Bayonne for a year or two weight losses were greater on boldly exposed specimens than on sheltered specimens. But weight loss-time curves soon crossed and from two years on weight losses were greater on sheltered specimens.

9. Pit depths did not tell exactly the same story as weight losses. Nickel steels had comparatively shallow pit depths and steels containing chromium comparatively deep pits. Pit depths seemed related to some extent to the number of points of attack. Pitting factors were decreasing with time. For thin steels pit depths are more important than weight losses.

10. The washing action of rain, while generally beneficial, may localize attack by concentrating corrosives at catchment areas. This accounted for the severe corrosion of certain edges of the Block Island specimens.

11. Variations in wetness and atmospheric pollution affect the corrosion behavior. At Block Island the corrosivity of the atmosphere has been increasing. This was probably connected with an increasing amount of fog and with an increasing percentage of northerly winds bringing in industrial contamination.

REFERENCES

(1) H. S. Rawdon, "Atmospheric Corrosion Testing," Symposium on Corrosion Testing Procedures, p. 32, Chicago Regional Meeting, Am. Soc. Testing Mats. (1937). (Symposium issued as separate publication.)

(2) H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 554 (1945).

(3) N. B. Pilling and W. A. Wesley, "Atmospheric Durability of Steels Containing Nickel and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 643 (1940).

(4) F. L. LaQue, Unpublished Data, The International Nickel Co., Inc.

(5) H. J. French and F. L. LaQue, "Alloy Constructional Steels," Am. Soc. Metals (1942).

(6) C. P. Larrabee, "Atmospheric Corrosion of Iron," Corrosion Handbook, p. 120, John Wiley and Sons, Inc., New York (1948).

(7) W. O. Binder and C. M. Brown, "At-

mospheric Corrosion of High Chromium Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 593 (1946).

(8) C. P. Larrabee, "Corrosion of Steels in Marine Atmospheres and in Sea Water," *Transactions*, Electrochem. Soc., Vol. 87, p. 161 (1945).

(9) E. S. Taylerson, "Atmospheric Exposure Tests on Copper Bearing and Other Irons and Steels in the United States," *Journal*, Iron and Steel Inst., Vol. 143, No. I, p. 287 (1941).

(10) W. H. J. Vernon, "Laboratory Study of Atmospheric Corrosion of Metals, Part III, Secondary Product or Rust (Influence of SO_2, CO_2, and Suspended Particles on Rusting of Iron)," *Transactions*, Faraday Soc., Vol. 31, p. 1678 (1935).

(11) J. C. Hudson, "Present Position of the Corrosion Committee Field Tests on Atmospheric Corrosion," *Journal*, Iron and Steel Inst., Vol. 148, No. II, p. 161 (1943).

(12) H. R. Copson, Discussion of Reference 8, *Transactions*, Electrochem. Soc., Vol. 87, p. 181 (1945).

(13) H. Pray and J. L. Gregg, "An Accelerated Atmospheric Corrosion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 758 (1941).

(14) J. A. Jones, "Effects of Phosphorus on Mechanical and Corrosion Resisting Properties of Low-Carbon, Low-Alloy Structural Steels," *Journal*, Iron and Steel Inst., Vol. 135, No. I, p. 113 (1937).

(15) S. C. Britton, "Some Properties of Commercial Steel Sheets Containing Additions of Copper, Manganese, Chromium, and Phosphorus," *Journal*, Iron and Steel Inst., Vol. 135, No. I, p. 161 (1937).

(16) W. H. J. Vernon, Discussion of Reference 2, *Proceedings* Am. Soc. Testing Mats., Vol. 45, p. 586 (1945).

(17) C. P. Larrabee, "The Effect of Specimen Position on Atmospheric Corrosion Testing of Steel," *Transactions*, Electrochem. Soc. Vol. 85, p. 297 (1944).

(18) G. Schikorr, "Uber das Atmospharische Rosten des Eisens II," *Zeitschrift Electrochem.*, Vol. 43, p. 697 (1937).

(19) W. A. Wesley and H. R. Copson, "Weathering Behavior of Corrosion-Resistant Steel Insect Screens," *Proceedings*, Am. Soc., Testing Mats., Vol. 46, p. 652, (1946).

(20) H. Baudot and G. Chaudron, "Essais de Corrosion Naturelle de Longe Duree sur Divers Aciers de Constuction dans l'Air Atmosphere et dans Differenates Eaux de Mer et de Riviere," *Rev. de Metallurgie*, Vol. 43, pp. 3, 19 (1946).

(21) K. F. Daeves, K. F. Mewes, and E. H. Schulz, "Significance and Evaluation of Atmospheric Corrosion Tests on Carbon and Low Alloy Steels," Korrosion u. Metallschutz, Vol. 19, p. 233 (1943).

(22) R. F. Passano, "The Harmony of Outdoor Weathering Tests," Symposium on Outdoor Weathering, p. 28, Washington Regional Meeting, Am. Soc. Testing Mats. (1934). (Symposium issued as separate publication.)

(23) G. Schikorr, "Die Abhangigkiet der Prufung des Atmospharisches Rostens des Eisens von den Zufalligkeiten der Versuchausfuhrung," Korrosion u. Metallschutz, Vol. 16, p. 422 (1940).

(24) Report of Committee A-5, Subcommittee III, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, Part I, pp. 146–153 (1946).

(25) A. M. Portevin, Discussion of Reference 15, *Journal*, Iron and Steel Inst., Vol. 135, No. I, p. 178 (1937).

(26) Report of Committee B-3, Subcommittee VII on Atmospheric Corrosion Tests of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 225 (1944).

(27) G. N. Schramm and E. S. Taylerson, "The Influence of Rainfall and Smoke on the Corrosion of Iron and Steel," Symposium on Outdoor Weathering, p. 51, Washington Regional Meeting, Am. Soc. Testing Mats. (1934). (Symposium issued as separate publication.)

(28) G. Schikorr, "Uber das Atmospharische Rosten des Eisens," *Zeitschrift Electrochem.*, Vol. 42, p. 107 (1936).

DISCUSSION

Mr. F. L. La Que[1] *(presented in written form)*.—The principal conclusions reached by Copson are supported so well by the data he has presented that additional corroboration of them is probably unnecessary.

Nevertheless, it may not be out of place to emphasize that while the incidental conditions of exposure during a test may influence the extent of superiority of alloyed steels over unalloyed ones, the alloy steels never appear at a disadvantage. At least some benefit corrosionwise may be expected from their use, and in general their advantage over unalloyed steel increases with the severity of the environment as illustrated by the data in Table V.

The information provided by Copson may also serve as a guide to design and application so as to secure the maximum benefit from the use of alloy steels where extra corrosion resistance is desired.

Some data obtained recently from tests of open-hearth irons at Kure Beach supplement those given in the paper with respect to effects of position of test specimens and their distance from the ocean. One of the objects of this test was to compare corrosion of specimens exposed vertically with that of specimens exposed at an angle of 30 deg. from the horizontal. Vertical exposure is practiced by J. C. Hudson—investigator for the British Iron and Steel Institute—in connection with his studies of atmospheric "pollution" as indicated by corrosion of ingot iron at several test stations. Inclined exposure of steel specimens is standard practice in this country.

One of the materials used was an ingot iron of the following composition:

Carbon, per cent	0.02
Sulfur, per cent	0.020
Manganese, per cent	0.04
Silicon, per cent	0.002
Phosphorus, per cent	0.004
Copper, per cent	0.008

The vertical specimens measured 2 by 4 by $\frac{1}{8}$ in. and the inclined specimens measured 4 by 6 by $\frac{1}{8}$ in. One set of specimens was exposed 80 ft. from the ocean and a duplicate set 800 ft. from the ocean. The duration of exposure was one year. After exposure the specimens were cleaned for reweighing by treatment as cathodes in warm inhibited sulfuric acid. Results are summarized in the following table:

EFFECT OF ANGLE OF EXPOSURE ON CORROSION OF OPEN-HEARTH IRON IN MARINE ATMOSPHERES. DURATION OF EXPOSURE 1 YR.

Angle of Exposure	Weight Loss, g. per sq. dm.		Average Pit Depth, in.	
	80 ft. from Ocean	800 ft. from Ocean	80 ft. from Ocean	800 ft. from Ocean
Vertical	91.3	15.4	0.021	0.017
Inclined 30 deg. from horizontal	64.0	13.0	0.024	0.018

These data with respect to effect of position of exposure are in line with those reported previously by Taylerson for rural and industrial atmospheres.[2] It is evident that weight loss is greater for specimens exposed vertically, but that pitting is less sensitive to the angle of exposure.

---

[1] In Charge of Corrosion Engineering Section, Development and Research Division, The International Nickel Company, Inc., New York, N. Y.

[2] C. S. Taylerson, "Atmospheric Exposure Tests on Copper Bearing and Other Irons and Steels in the United States," *Journal*, Iron and Steel Inst., Vol. 143, No. I, p. 287 (1941).

It is in order also to draw attention to the fact already illustrated by Copson's Table V that the corrosion of some materials may be influenced violently by their actual distance from the ocean within what might be called an area "near" the ocean. From this it follows that all "marine" atmospheres are not alike and that such a description should be qualified by data referring to the corrosion of some key materials used for calibration purposes if maximum benefit is to be obtained from tests at "marine" corrosion test stations and if comparison of results from different stations is to be facilitated. In this connection, it may be noted that a start along this line is being made by Subcommittee VII on Weather of A.S.T.M. Committee B-3 which is exposing standard specimens of steel and zinc for calibration purposes at several of the A.S.T.M. atmospheric corrosion testing stations.

MR. H. R. COPSON (*author's closure*). —The discussion by F. L. La Que calls for little comment, except perhaps with regard to the exposure of key materials for calibration purposes. This is a good move and nothing better suggests itself, but the difficulty is to select the key materials and to interpret the results accurately. In the paper it was shown that the results obtained varied not only with the location, but also with the composition of the steel and with the duration of the tests. It follows that comparisons based on key materials will apply only to the key materials themselves and for the lengths of exposure actually used.

This can be illustrated by a comparison between identical steels exposed at Block Island and on the rack at Kure Beach 800 ft. from the ocean. For the better low-alloy steels the corrosion rates were practically identical at the two locations. On the other hand, fast corroding ingot irons lost about 25 per cent more weight at Block Island. If pure ingot iron was selected as the key material, it might be concluded that conditions were 25 per cent more corrosive at Block Island, whereas if a good low-alloy steel was selected as the key material, it might be concluded that there was no significant difference between the two locations. Neither conclusion would tell the whole truth.

The same point is illustrated in Table V, which gave a comparison at Kure Beach between racks 80 ft. and 800 ft. from the ocean. Fast corroding steels were more affected by nearness to the ocean than the better low-alloy steels.

It perhaps should be emphasized that different metals and alloys behave differently. Thus 5-yr. weight losses on zinc were higher at Bayonne that at Block Island, which was the reverse of the behavior of steel. It follows that calibration results on one metal, such as steel, should not be extended to another metal, such as zinc, and that if both steel and zinc are used for calibration purposes, the results should not be extended to other metals.

ATMOSPHERIC DURABILITY OF STEELS CONTAINING NICKEL AND COPPER—ADDITIONAL EXPOSURE DATA*

By N. B. Pilling[1] and W. A. Wesley[1]

Synopsis

The prior publication (**1**)[2] on this subject appeared in 1940 and described six series of weathering tests designed to evaluate the durability of sheet specimens of low-alloy steels containing nickel and copper. The present communication brings up to date the published results of those exposure tests which were continued beyond 1940. In the oldest series, observations at the end of 22 yr. exposure are now made available. In the series in which large sheet specimens simulate some of the conditions encountered in roofing service, the advantage of nickel-copper steels over copper steels is becoming more impressive as the years pass by. Long-continued tests show that it is more significant to compare steels on the basis of how long a time is required to arrive at a given unit weight loss than to compare them on the basis of weight loss suffered in a fixed period of time.

The beneficial effects of nickel upon the weathering resistance of steel increase with increasing nickel content. Addition of copper to a nickel steel improves the life, but the optimum amount is less than the amount of nickel and remains well below 2 per cent even with a 4 per cent nickel content. With long exposure periods, there is evidence that not only phosphorus but also silicon additions to nickel-copper steels have a beneficial effect on behavior in the industrial atmosphere. Effects of manganese and carbon are unimportant in this type of atmosphere.

There was a paucity of data in the literature prior to 1940 on the effect of nickel in the low-alloy range on atmospheric corrosion of steel, but since that time this subject has received increasing attention. Taylerson (**2**) included a $3\frac{1}{2}$ per cent nickel steel in a study of copper steels exposed at one rural and two industrial atmospheres. Weight loss *versus* time curves were presented for a 5-yr. exposure period, which demonstrated that the advantage of the nickel steel over $\frac{1}{4}$ per cent copper steels increased with duration of test in accord with the authors' earlier publication (**1**). In developing an accelerated atmospheric corrosion test, Pray and Gregg (**3**) obtained weight loss data for 1 and 2 yr. exposure of a series of 0.6 per cent chromium-0.6 per cent copper steels containing various amounts of nickel from 0.7 to 3.0 per cent. Calling the weight loss of a simple $\frac{1}{4}$ per cent nickel steel 100 per cent, the complex steels lost weight in 2 yr. to a relative extent of 67 per cent for a 0.76 per cent nickel content and 48 per cent for the 3 per cent nickel alloy. This spread would undoubtedly have been greater had the weathering tests continued longer.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Director and Assistant Director, respectively, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 617.

Data originating with the Carnegie-Illinois Steel Corp. were made available in the form of a curve showing the decrease in loss in weight with increasing nickel content of a set of steels exposed for 5 yr. to an industrial atmosphere (4). The beneficial effect of nickel persisted to the maximum level of nickel content represented, namely, 5 per cent. Some time-corrosion curves for 5 per cent nickel steels are included in a paper by Larrabee (5) from tests covering 8 yr. of exposure in an industrial atmosphere. The shape of these curves confirms an earlier conclusion (1) that the rate of corrosion of nickel steels continues to decrease with lapse of time. These results are further substantiated by another Larrabee publication (6) containing additional loss in weight-time curves for $3\frac{1}{2}$ per cent nickel steels and a nickel-copper steel. As was concluded in the earlier paper by the present authors (1), the advantage of the nickel and nickel-copper steels over plain copper steel was even more pronounced after a few years in a marine atmosphere than in the industrial atmospheres.

A mass of data on low-alloy constructional steels was presented by Copson (7) in developing a theory of the mechanism of rusting of low-alloy steels in the atmosphere. Further results from the same series of exposure tests are to be found in a later paper (8). An important conclusion of this work is that whereas copper and chromium additions to steel were more effective in the industrial than in the marine atmosphere, and manganese was more effective in the marine than in the industrial, nickel additions were effective in lowering corrosion rates at both test sites. Another significant result was the substantiation of the idea that it takes years of exposure for the full benefits of alloying to be realized in the form of reduction of rate of weathering.

TABLE I.—WEATHERING TESTS BEGUN IN 1925.

Type[a]	Chemical Composition, per cent					Life to First Perforation, yr.			
	Nickel	Copper	Manganese	Silicon	Carbon	Rural Atmosphere	Marine Atmosphere	Moderate Industrial Atmosphere	Heavy Industrial Atmosphere
Ingot iron	...	...	0.21	0.03	0.04	11	1.5	1.5	1.5
Copper steel (commercial)	...	0.17	...	...	0.07	14.5	1.5	3.0	2.1
2 per cent nickel-copper steel	1.39	0.61	0.36	0.14	0.07	14.5	over 5	4	3.2
4 per cent nickel-copper steel	2.76	1.18	0.22	0.08	0.11	over 17.5	over 5	6	10
4 per cent nickel-copper steel	2.8	1.2	...	...	0.1	over 17.5	over 5	10	14.5
6 per cent nickel-copper steel	4.2	1.8	0.38	0.13	0.08	over 22.5	5	8	over 20.7
8 per cent nickel-copper steel	5.42	2.36	0.40	0.14	0.08	over 22.5	over 5	18.5	over 20.7
10 per cent nickel-copper steel	6.95	3.03	0.36	0.14	0.18	over 22.5	over 5	8[b]	over 20.7
10 per cent copper-nickel steel	3.0	6.71	0.40	0.18	0.05	over 22.5	3.5	5.5	12

[a] Thickness of specimens, 0.020 in.
[b] Failed by cracking.

WEATHERING TESTS BEGUN IN 1925

The most recent information on this old exposure of experimental steels is shown in Table I where perforation of sheet by corrosion at a distance of $\frac{1}{4}$ in. or more from the edges is considered the criterion of failure. Corrosion of the steels containing more than 2 per cent nickel plus copper is so slow at the rural test site in West Virginia that it will be amazing if the site can be maintained intact until all the specimens have failed

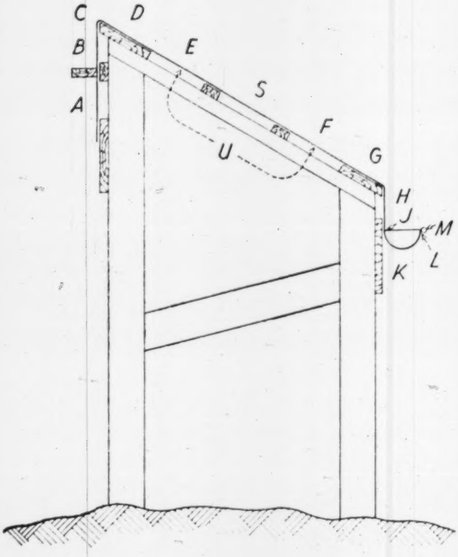


FIG 1.—End View of Simulated Roofing Test Specimen.

(*A*) Vertical surface, partly sheltered from direct impingement of rain.
(*B*) Vertical surface, boldly exposed to rain.
(*C*) Sharp bend in sheet.
(*D*) Area having lower surface in contact with wood.
(*E*) and (*F*) Plane inclined surfaces.
(*G*) Area having lower surface in contact with wood.
(*H*) Vertical surface, boldly exposed.
(*J*) Area of water-line attack when trough is full of rain water.
(*K*) Area completely submerged when trough is full of rain water.
(*L*) Area of water-line attack.
(*M*) Trough-bead.
(*S*) Lock seam, one-half soldered, one-half not soldered.
(*U*) Fully sheltered lower surfaces.

by corrosion. The passing of the years confirms the conclusion drawn in the previous report (1) that the nickel-copper steels consistently demonstrate superiority over the copper steels in all atmospheres represented.

SIMULATED ROOFING TESTS

Figure 1 showing a sketch of the roofing test specimens, and Table II giving the analysis of the materials tested, are repeated from the original publication for convenience in studying the results of inspection. In Table III the records begun in 1939 are brought up to date. In recent years the inspections have been made approximately every 12 months.

With the more resistant steels, an appreciable difference in life is becoming evident between the areas of sheet boldly exposed to the rain and area *A* where both surfaces of the sheet are protected from rain. In most cases, failure is occurring sooner at area *A* in accord with other published data (5, 8). The same sort of effect may be operating to lengthen the life of the sloping areas *C* to *G* in comparison with the verticle

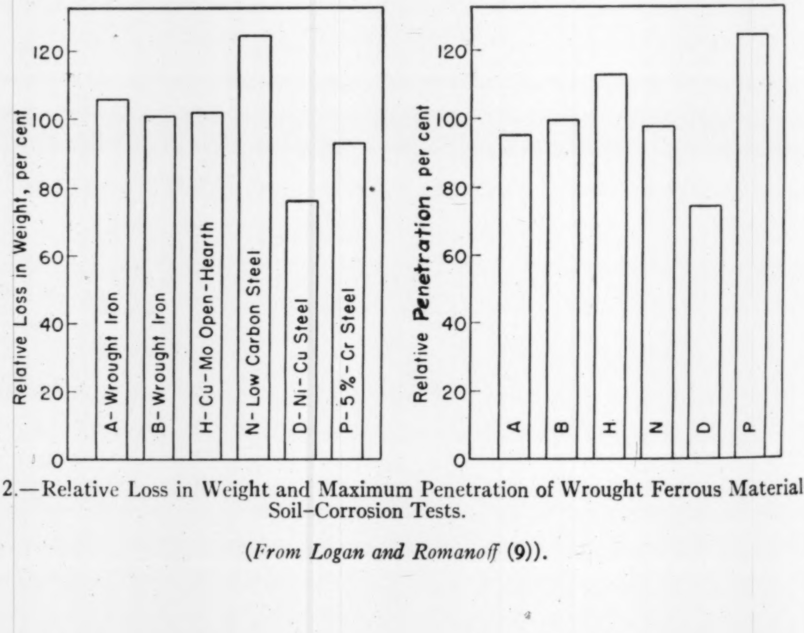


FIG. 2.—Relative Loss in Weight and Maximum Penetration of Wrought Ferrous Materials in Soil-Corrosion Tests.

(*From Logan and Romanoff* (9)).

TABLE II.—ANALYSIS OF MATERIALS IN SIMULATED ROOFING TESTS.

Type of Steel	Analysis, per cent						
	Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Sulfur
Commercial:							
Bessemer steel		0.008	0.40	0.005	0.095	0.111	0.059
Ingot iron		0.06	0.02	0.001	0.015	0.008	0.026
Open-hearth copper steel		0.200	0.39	0.005	0.017	0.006	0.018
Rephosphorized open-hearth copper steel		0.252	0.49	0.005	0.065	0.052	0.022
Bessemer copper steel		0.212	0.40	0.018	0.100	0.112	0.059
Copper-molybdenum iron[a]		0.46	0.12	0.002	0.009	0.015	0.026
Experimental:							
2% nickel steel	1.96	0.03	0.14	0.14	0.06	0.013	0.028
1% nickel, 1% copper	1.05	0.87	0.17	0.13	0.05	0.014	0.031
1.5% nickel, 1% copper	1.58	0.86	0.32	0.20	0.05	0.043	0.068
2% nickel, 1% copper	1.77	0.93	0.13	0.13	0.05	0.012	0.030
1% nickel, 2% copper	1.24	2.23	0.39	0.63	0.08	0.017	0.033
2% nickel, 2% copper	2.16	1.97	0.28	0.52	0.06	0.018	0.005
3% nickel, 1% copper	3.12	1.14	0.20	0.21	0.08	0.015	0.009
3% nickel, 1.3% copper	2.82	1.30	0.14	0.41	0.06	0.015	0.027
3% nickel, 2% copper	2.78	1.83	0.27	0.20	0.06	0.013	0.008
4.4% nickel, 2% copper	4.40	2.05	0.24	0.20	0.11	0.019	0.008
5.5% nickel, 1.3% copper	5.53	1.31	0.17	0.15	0.08	0.012	0.007
6.4% nickel, 1% copper	6.35	1.06	0.16	0.31	0.08		
6.4% nickel, 2% copper	6.40	2.12	0.13	0.12	0.11	0.014	0.011

[a] Contains molybdenum, 0.09 per cent.

TABLE III.—SIMULATED ROOFING TESTS.

Time of initial failure at different areas of each specimen. Blank spaces mean no failure at time of last inspection. Letters refer to areas designated in Fig. 1. Results show life in months. Average initial thickness of sheets was 0.033 in.

Type of Steel	Rural Atmosphere					Marine Atmosphere[a]					Industrial Atmosphere					Sulfurous Atmosphere				
	A	B	C to G	K	J and L	A	B	C to G	K	J and L	A	B	C to G	K	J and L	A	B	C to G	K	J and L
Bessemer 0.01% Cu, 0.11% P	74	119	62	15	25	15[b]	15[b]	15[b]	15[b]	15[b]	18	18	25	12	12	12	12	12	12	12
Ingot iron 0.06% Cu, 0.01% P				62	74	25	61		15	15	130	130	178	29	25	42	58	34	22	12
Open-hearth copper, 0.20% Cu, 0.01% P				25	74	37	61		25	25	130	130	178	25	25	58	58	58	22	12
Rephosphorized open-hearth copper, 0.25% Cu, 0.05% P				62	94	25			15	15	130	130		29	26	58	73	84	34	22
Bessemer copper, 0.21% Cu, 0.11% P				62	133	25	61		25	25	136	142		38	38	73	73	84	34	12
Copper-molybdenum iron, 0.46% Cu, 0.01% P				62	94	25			15	15	136	136		29	29	58	73	84	22	22
2% Ni, 0% Cu				62	94				25	25	136	168		29	26	58	84	176	34	22
1% Ni, 1% Cu				62	94	61			37	30	136			29	26	73	103		34	22
1.5% Ni, 1% Cu				62	74				37	30	136			29	29	84	103		34	22
2% Ni, 1% Cu				62	74				25	37	136			29	29	73	103		34	22
1% Ni, 2% Cu				62	94				37	30	136	168		29	26	73	84		34	22
2% Ni, 2% Cu				62	94				37	30	136			29	26	84	151		34	22
3% Ni, 1% Cu				62	94				37	37				29	29				34	34
3% Ni, 1.3% Cu				62	94				37	45	168			29	38	113			34	34
3% Ni, 2% Cu				62	94				37	37				38	29				34	34
4.4% Ni, 2% Cu				62	119				37	37				38	29				34	34
5.5% Ni, 1.3% Cu				62	119				37	61				29	26				34	34
6.4% Ni, 1% Cu				62	119				45	45				45	38				42	34
6.4% Ni, 2% Cu				62	119				45	45				38	29				34	34

[a] Testing station destroyed by hurricane at end of 75 months. Last inspection made at 73 months.

[b] Condition indicated that failure occurred in considerably less than 15 months.

TABLE IV.—EFFECTS OF COMPOSITION VARIABLES, 1933 SERIES.

Minor Elements, per cent				Life of 0.015-in. Strips, at Bayonne, N. J., Months												Average of All Compositions
Nickel, per cent				0	0	0	0	1	1	2	2	2	4	4	4	
Copper, per cent				0	½	1	2	0	½	0	1	2	0	1	2	
Mn	Si	C	P													
0.3	0.1	0	0	14	24	31	35	49	37	31	102	59	59	96	72	51
0.6	0.1	0	0	[a]	15	24	15	26	59	31	37	35	96	144	90	52
1.0	0.1	0	0	35	46	59	28	28	31	37	37	37	90	102	84	51
2.0	0.1	0	0	[a]	26	52	31	31	63	72	84	[a]	[a]	[a]	[a]	51
0.3	0.2	0	0	19	26	31	31	28	37	37	44	40	90	102	115	50
0.3	0.5	0	0	24	[a]	28	31	44	31	[a]	72	59	115	126	115	65
0.3	1.0	0	0	28	28	31	[a]	46	35	37	[a]	59	131	157	157	71
0.3	0.1	0.2	0	[a]	102	31	31	28	31	40	63	44	72	126	102	61
0.3	0.1	0.3	0	26	63	28	37	59	28	31	72	37	102	128	115	61
0.3	0.1	0.4	0	[a]	26	31	52	31	115	25	79	44	79	128	102	65
0.3	0.1	0	0.03	[a]	[a]	59	52	37	37	44	59	59	72	102	102	62
0.3	0.1	0	0.06	28	[a]	46	72	96	90	131	46	[a]	90	115	102	82
0.3	0.1	0	0.09	28	[a]	59	44	102	37	40	84	171	180	102	102	86
0.3	0.1	0	0.12	44	[a]	40	28	37	44	40	115	44	126	180	102	73
				...	...	...	...	...	...	...	...	...	...	...	...	...
Average life				27	39.5	39	37	46	48	46	69	57	100	124	105	59

[a] This composition not represented, due to loss or error.

TABLE V.—OTHER RESULTS FROM 1933 SERIES.

Specimens of the steels from the Roofing Test of Table II were rolled to 0.015 in. gage and were exposed at same time as experimental steels of Table IV. Atmospheric exposure at Bayonne, N. J.

Type of Steel	Life, months
Bessemer 0.01% Cu, 0.11% P	12
Ingot iron 0.06% Cu, 0.01% P	24
Open-hearth copper 0.20% Cu, 0.01% P	35
Rephosphorized open-hearth copper 0.25% Cu, 0.05% P	42
Bessemer copper 0.21% Cu, 0.11% P	35
Copper-molybdenum iron 0.46% Cu, 0.01% P	31
2% Ni, 0% Cu	59
1% Ni, 1% Cu	46
1.5% Ni, 1% Cu	59
2% Ni, 1% Cu	59
1% Ni, 2% Cu	37
2% Ni, 2% Cu	72
3% Ni, 1% Cu	171
3% Ni, 1.3% Cu	115
3% Ni, 2% Cu	More than 180
4.4% Ni, 2% Cu	171
5.5% Ni, 1.3% Cu	More than 180
6.4% Ni, 1% Cu	More than 180
6.4% Ni, 2% Cu	More than 180

unsheltered area *B*, with better rinsing by rain of the sloping surfaces. The difference in this case is more pronounced to date in the nickel-copper steels than in the plain copper steels and should prove advantageous to the former in actual roofing service.

SOIL CORROSION TESTS

Additional data are now available from the soil corrosion studies of the National Bureau of Standards which were begun in 1931. The results obtained on tubular wrought ferrous specimens have been neatly summarized by Logan and Romanoff (**9**) in Fig. 2, which presents the relative average values of corrosion of all the materials at 10 locations for 2, 5, 7, and 9 yr. interment. It is evident that the nickel-copper steel D (2.47 per cent nickel and 1.08 per cent copper) has continued to be superior to the others, both in respect to weight loss and resistance to pitting.

Some flat plate specimens were later exposed at the same test sites in 1937. In this case, the tightly adherent mill scale on a nickel-copper steel was not removed prior to the burial of the test panels. The mill scale appeared to promote pitting so that the depth of penetration of the nickel-copper steel was about equal to that of the mild and copper steels at the end of 4 yr. of exposure.

EFFECT OF VARIABLES IN COMPOSITION (1933 SERIES)

This set of exposure tests can now be considered completed since all but four of the original thin strip specimens representing 168 different heats of low-alloy steels have failed by corrosion. The four remaining contain 5 per cent or more total nickel plus copper. Results are presented in Tables IV and V. All the steels of Table IV were prepared in a high-frequency induction furnace using an Armco iron base. The details of preparation and analysis can be found in the earlier report (1).

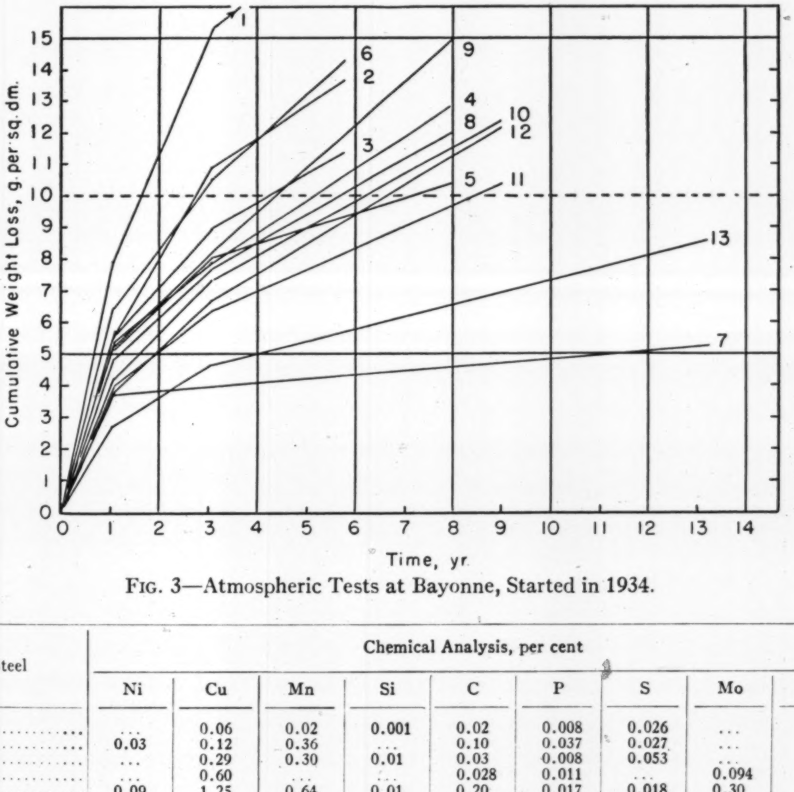


FIG. 3—Atmospheric Tests at Bayonne, Started in 1934.

Steel	Chemical Analysis, per cent								
	Ni	Cu	Mn	Si	C	P	S	Mo	Cr
No. 1	...	0.06	0.02	0.001	0.02	0.008	0.026	...	...
No. 2	0.03	0.12	0.36	...	0.10	0.037	0.027	...	...
No. 3	...	0.29	0.30	0.01	0.03	0.008	0.053	...	...
No. 4	...	0.60	...	...	0.028	0.011	...	0.094	...
No. 5	0.09	1.25	0.64	0.01	0.20	0.017	0.018	0.30	...
No. 6	...	...	0.52	0.15	0.21	0.017	0.024	0.22	...
No. 7	0.05	0.45	0.33	0.79	0.13	0.15	...	...	1.15
No. 8	0.48	0.50	0.46	0.01	0.12	0.011	0.029	...	0.41
No. 9	1.37	...	0.70	0.91	0.19	0.015	0.025	...	...
No. 10	2.07	...	0.37	...	0.07	0.011	0.029	...	...
No. 11	1.58	0.86	0.32	0.20	0.05	0.045	0.069	...	...
No. 12	1.74	0.09	0.36	0.20	0.125	0.018	0.025	0.24	...
No. 13	5.03	...	0.49	0.30	0.13	0.018	0.019	...	...

The rather erratic results to be seen if small groups of specimens close together in Table IV are compared was the subject of a study by H. R. Copson of this laboratory. Although the data are presented as time to disintegration of the edges, the weights of all specimens had been recorded before they were exposed. It was found that there was sufficient correlation between initial weights of specimens and the life data within adjacent groups to conclude definitely that the variation in initial thickness of these experimentally rolled strips was a major cause of variation in observed life. The other conclusion drawn from this study was that more reproducible and consistent results could have been obtained by determining weight losses with thicker specimens exposed in replicate, which permits removal of rust to detect perforation otherwise invisible.

Effects of Manganese, Silicon, Carbon, and Phosphorus:

At the earlier stage of this study, it was concluded (1) that the manganese, silicon, and carbon contents of the steels had no influence upon the life of the specimens in this industrial atmosphere. Examination of the averages of horizontal columns in Table IV now tends to confirm the lack of effect of manganese but shows a significant beneficial influence of silicon as well as a faint improvement with carbon. There is no question about the improvement contributed by addition of phosphorus to these nickel-copper steels.

Effects of Nickel and Copper:

The averages of the vertical columns of Table IV make clear the great increase in life with change in nickel and copper content from left to right. Addition of copper to a nickel steel increases its resistance to weathering, but the maximum effect is obtained with something less than 2 per cent copper, even in the presence of 4 per cent nickel.

ATMOSPHERIC TESTS STARTED IN 1934

Two sets of specimens of this group of alloy constructional steels were removed from the atmospheric exposure racks at Bayonne at the end of 1 and 3 yr., respectively, as described in the earlier report (1). It was later decided that less information would be secured if all the specimens of the third set were removed at the same time than if the more resistant steels were permitted to remain longer. It does not seem as important to know what the relative losses in weight of a series of steel were at a given date as to know how many years would pass before each steel suffered a given weight loss.

The results are presented in Fig. 3 where a dotted line is shown representing a weight loss of 10 g. per sq. dm. per specimen. Experience in atmospheric corrosion tests of steels run by this laboratory has shown that perforation of steel sheet 0.03-in. thick is apt to occur when the loss in weight of chemically cleaned specimens reaches approximately this level. This initial perforation can be detected only by removing the rust from the specimen. In Table VI, the various steels of the 1934 series are arranged in order of the time of exposure elapsed until a loss of 10 g. per sq. dm. occurred. This time was estimated from the corrosion curves and represents great uncertainty of extrapolation for the very resistant steels Nos. 7 and 13.

TABLE VI.—YEARS REQUIRED FOR A WEIGHT LOSS OF 10 G. PER SQ. DM.

Steel	Years	Steel	Years
No. 1	1.6	No. 10	6.3
No. 2	2.7	No. 12	6.6
No. 6	2.8	No. 5	7.1
No. 3	4.2	No. 11	8.6
No. 9	4.3	No. 13	(16)
No. 4	5.1	No. 7	(>>16)
No. 8	5.7		

When the exposure specimens of steels Nos. 7 and 13 were cleaned at the end of the test (13.25 yr.), it was observed that the maximum penetration by pitting of the copper-silicon-chromium steel was about 18 mils, whereas that of the 5 per cent nickel steel was only 12.3 mils. The pitting factor[3] of steel No. 7 was 6.8 as compared with the more normal 2.8 for steel No. 13.

---

[3] Pitting Factor = the depth of the deepest pits resulting from corrosion divided by the average penetration as calculated from weight loss.

It can be concluded from this series of tests that good resistance to weathering in an industrial atmosphere is to be found in the more highly alloyed constructional steels and the copper-molybdenum and copper-silicon chrome steels.

Acknowledgment:

Many of the inspections of these exposure series were made by H. R. Copson of this laboratory, whose assistance is gratefully acknowledged.

REFERENCES

(1) N. B. Pilling and W. A. Wesley, "Atmospheric Durability of Steels Containing Nickel and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 643 (1940).

(2) E. S. Taylerson, "Atmospheric Exposure Tests on Copper Bearing and Other Iron and Steels in the United States," *Journal*, Iron and Steel Inst., Vol. 143, No. I, p. 287 (1941).

(3) H. Pray and J. L. Gregg, "An Accelerated Atmospheric Corrosion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 758 (1941).

(4) H. J. French and F. L. LaQue, "Alloy Constructional Steels," Cleveland p. 173, (1942).

(5) C. P. Larrabee, "The Effect of Specimen Position on Atmospheric Corrosion Testing of Steel," *Transactions*, Electrochemical Soc., Vol. 85, p. 297 (1944).

(6) C. P. Larrabee, "Corrosion of Steels in Marine Atmospheres and in Sea Water," *Transactions*, Electrochemical Soc., Vol. 87, p. 161 (1945).

(7) H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 554 (1945).

(8) H. R. Copson, "Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels," see p. 591 of this publication.

(9) K. H. Logan and M. Romanoff, "Soil-Corrosion Studies, 1941," *Journal of Research* Nat. Bureau of Standards, Vol. 33, p. 145 (1944).

LABORATORY CORROSION TESTS OF IRON AND STEEL PIPES*

By G. A. Ellinger,[1] L. J. Waldron,[2] and S. B. Marzolf[3]

Synopsis

Continuous flow laboratory corrosion tests of ten types of uncoated ferrous pipes were made over periods of time extending up to ten years. The corroding medium was Washington, D. C., tap water continuously circulated through columns of the test samples. Corrosion was measured as loss of weight and depth of pits in relation to the time of exposure. While most of the pipes corroded at somewhat similar rates, there were small but real differences in the corrosion of some of the materials.

Continuous-flow laboratory-corrosion tests of steel pipe, sponsored by the Republic Steel Corp. through the National Research Council, were started at the National Bureau of Standards in 1933. Only two compositions of steel pipes were included and each specimen was exposed for only an 8-week period. The results of these tests[4] were affected to a considerable extent by turbulent flow and other uncontrolled factors associated with the testing apparatus, and consequently were not conclusive. Since considerable interest was expressed by several agencies in extended pipe-corrosion tests, a new and more comprehensive investigation was undertaken by the National Bureau of Standards. This investigation was designed to test different types of commercially available pipes for a minimum of 3 yr. Specimens were to be removed from time to time and a sufficient number was to be included for extension of the test for as long as appeared to be desirable, perhaps up to 10 yr. To improve upon the earlier test results, changes were made in the apparatus to permit smooth non-turbulent flow of water through the specimens. The new tests were started in June, 1935, and, except for minor shutdowns for repairs or removal of specimens, ran continuously until June, 1945. At one time or another during the 10-yr. period, various individuals were associated with the test. It was initially directed by H. S. Rawdon. L. J. Waldron assembled the equipment, started the test, and conducted it during the first and last four years. In the interim, the experimental work was conducted by B. J. Baecher and T. D. Brown.

Materials and Method

The materials investigated were sections of (*a*) eight wrought pipes, three of these being made of open-hearth steel, two of ingot iron, and three of wrought iron; and (*b*) two cast-iron pipes, one

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Chief, Optical Metallurgy Section, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[2] Chemist, Naval Research Laboratory, Washington D. C.

[3] Metallurgist, National Bureau of Standards, Washington, D. C.

[4] H. S. Rawdon and L. J. Waldron, "Continuous-Flow Corrosion Tests of Steel Pipe," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 233 (1935).

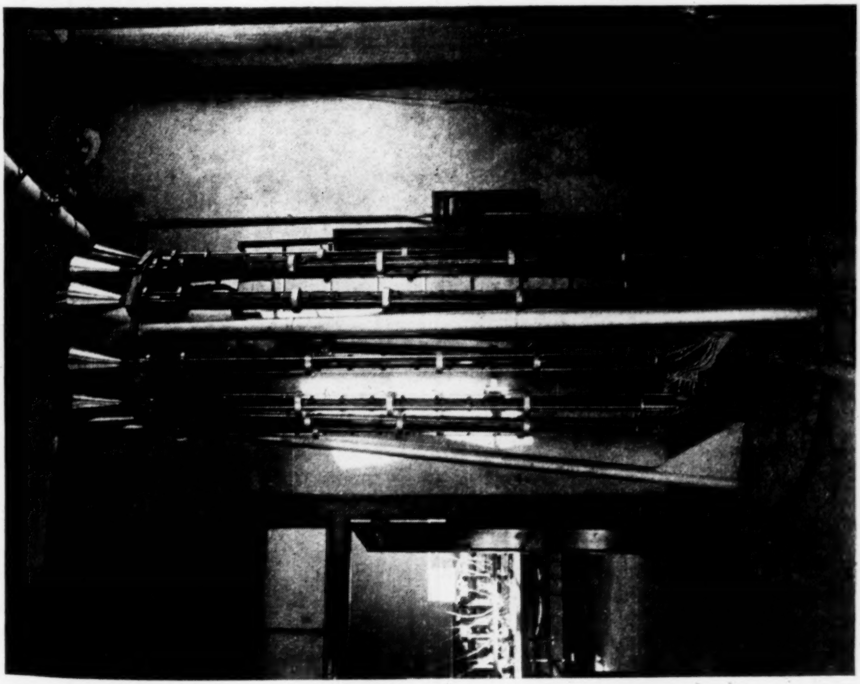


FIG. 1.—Test Apparatus Showing the Columns of Specimens in Place.

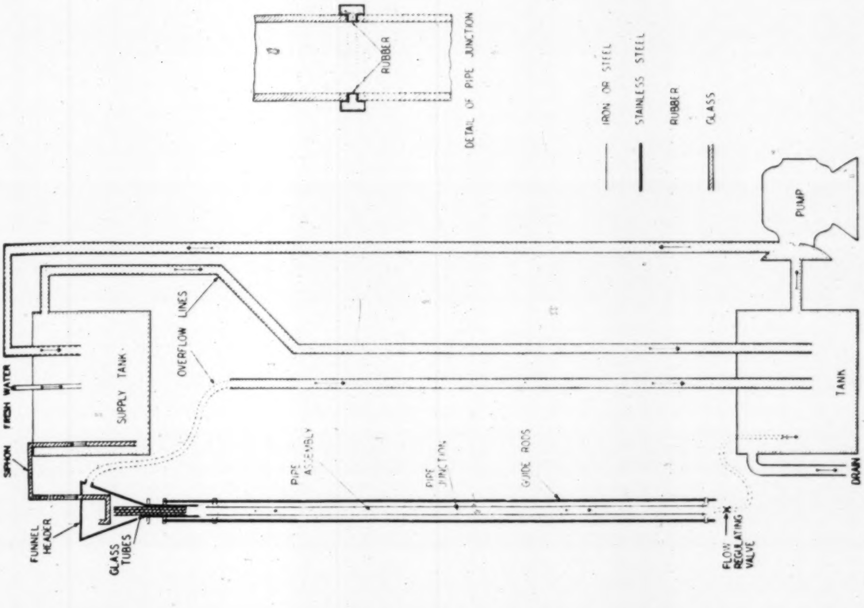

SIPHON
FRESH WATER
SUPPLY TANK
OVERFLOW LINES
FUNNEL HEADER
GLASS TUBES
PIPE ASSEMBLY
PIPE JUNCTION
GUIDE RODS
FLOW REGULATING VALVE
TANK
DRAIN
PUMP
RUBBER
DETAIL OF PIPE JUNCTION
IRON OR STEEL
STAINLESS STEEL
RUBBER
GLASS

FIG. 2.—Sketch Showing the Details of Assembly and Operation of the Test Apparatus.

centrifugally cast, the other sand cast. The identification symbols and chemical compositions are given in Table I.

The wrought pipes were of standard weight, with a nominal 2-in. diameter and a wall thickness of about $\frac{3}{16}$ in. The cast-iron pipes had slightly thicker walls, that is about $\frac{1}{4}$ in., and slightly smaller internal diameters.

The method used was that of circulating Washington, D. C., tap water through a system of columns built up of test sections of these pipes. A photograph of the apparatus with all of the columns in place is shown in Fig. 1. The details of construction and the plan of operation are shown in Fig. 2.

TABLE I.—IDENTIFICATION SYMBOL AND COMPOSITION OF MATERIALS.[a]

Identification Symbol	Material	Composition, per cent									
		C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Graphite
A	Wrought iron—aston process	0.02	0.04	0.092	0.025	0.18	0.03				
B	Wrought iron—hand puddled	0.02	0.06	0.16	0.012	0.12	0.04				
C	Cast iron—centrifugal cast	3.61	0.64	0.34	0.088	2.46	0.37	...	0.33	...	2.96
D	Cast iron—sand cast	3.57	0.54	0.57	0.071	2.26	0.15	...	0.09	...	2.97
E	Open hearth steel	0.18	0.43	0.010	0.017	0.18	0.05				
F	Copper bearing steel	0.11	0.42	0.007	0.022	0.19	0.29				
G	Ingot iron	0.02	0.03	0.005	0.026	0.01	0.05				
H	Cu-Mo ingot iron	0.03	0.14	0.008	0.012	0.01	0.45	...	...	0.07	...
I	Cu-Cr-P-Si steel	0.08	0.21	0.16	0.016	0.80	0.39	...	1.10	...	...
J	Nickel-bearing wrought iron	0.02	0.05	0.087	0.012	0.10	0.08	1.72	...	...	...

[a] Analyses made by Chemistry Division, National Bureau of Standards.

Specimens $5\frac{1}{2}$ to 6 in. long were cut from the pipes and the ends were machined smooth and parallel on a lathe. In order to remove all surface dirt, grease, and mill scale, the specimens were cleaned prior to testing, by the following operations:

(*a*) Washed in strong alkali or soap solution,

(*b*) Rinsed in water,

(*c*) Pickled in warm 10 per cent sulfuric acid solution containing a commercial inhibitor,

(*d*) Washed in clean soap solution,

(*e*) Brushed in hot running water, and

(*f*) Dried.

A bristle brush was used for removing surface dirt, but when this adhered tightly and was difficult to remove, steel wire brushing was employed. The cast-iron specimens were cleaned very carefully so as not to remove the casting surface; immersion in the acid was therefore brief and brushing was held to a minimum. Each of the specimens was weighed and measured prior to exposure.

Fourteen specimens were assembled to form each column, there being eight of these columns in the apparatus. The details of the method of assembling the columns are shown in Fig. 3. Metallic contact and subsequent galvanic action between adjacent specimens was prevented by the use of rings of hard rubber. The specimens were fitted securely into recesses machined in the separators and the entire column was made watertight by tightening three equally spaced external tie rods. Care was taken to secure junctions as smooth as possible, since any projection, crevice, or irregularity would interfere with the normal flow of the water and create turbulence and eddies, which might affect the results. Turbulence of the water entering the apparatus was prevented by the insertion of a number of parallel glass rods in the top of each column. Each column was supported in a vertical position and maintained in a watertight condition by the tie rods, guide and supporting sleeves, and stainless steel holders. The equipment was designed to permit the continuous exposure of the inner surfaces of all of the specimens in as identical a manner as possible to water of known composition and moving at a constant velocity. The water within the apparatus came in contact only with the metal specimens, rubber, glass, and stainless steel (18 per cent chromium-8 per cent nickel).

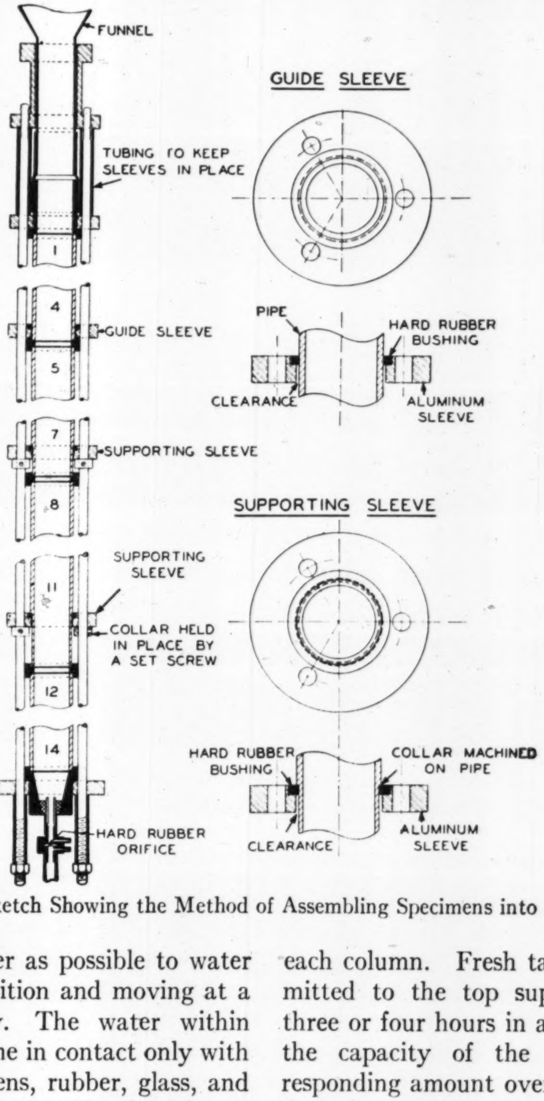

FUNNEL
TUBING TO KEEP SLEEVES IN PLACE
1
4
GUIDE SLEEVE
5
7
SUPPORTING SLEEVE
8
SUPPORTING SLEEVE
11
COLLAR HELD IN PLACE BY A SET SCREW
12
14
HARD RUBBER ORIFICE
GUIDE SLEEVE
PIPE
HARD RUBBER BUSHING
CLEARANCE
ALUMINUM SLEEVE
SUPPORTING SLEEVE
HARD RUBBER BUSHING
COLLAR MACHINED ON PIPE
CLEARANCE
ALUMINUM SLEEVE

FIG. 3.—Sketch Showing the Method of Assembling Specimens into a Column.

The capacity of the entire apparatus, including the top and bottom tanks and all columns and overflow pipes, was about 70 gal. The water circulation through the columns and overflow pipes was 40 to 50 gal. per min., and the water flow was maintained at a linear velocity of 30 ft. per min. by an adjustable orifice and manometer at the lower (outlet) end of each column. Fresh tap water was admitted to the top supply tank every three or four hours in amounts equal to the capacity of the system, a corresponding amount overflowing into the drain from the lower tank.

Six of the columns each contained specimens of only a single material, the seventh column contained specimens of materials I and J, while the eighth contained specimens of the two cast irons, materials C and D. Individual specimens were removed from each of the columns and were replaced with new ones of the same materials at the end of specified test periods, ranging in length up to 10 yr. The number of specimens exposed for each period, in months, is given in Table II.[5] The two cast irons, C and D, the Cu-Cr-P-Si steel I, and the nickel wrought iron J were exposed for a maximum of 7 yr., while all other materials were exposed for 10 yr.

TABLE II.—NUMBER OF SPECIMENS EXPOSED FOR EACH PERIOD.

Exposure Period, months	Materials A, B, E, F, G, H	Materials C, D	Materials I, J
6	3	2	2
12	2	3	2
18	1	..	..
24	1	1	1
30	1	..	..
36	2	2	2
42	1	1	1
48	3	2	2
54	1	..	..
60	3	2	2
72	5	4	3
84	2	1	2
120	3	..	..

It was necessary to protect the exterior surfaces of the specimens from corrosion during the life of the test. Originally all of the specimens were coated with a petroleum jelly-benzol mixture after installation, but because this could be easily removed accidentally an asphalt varnish was subsequently used for this purpose.

After removal from the test columns, the specimens were dipped in trichlorethylene to remove the varnish and then were cleaned free of corrosion products by the following operations:

(*a*) Brushed with a soft bristle brush under water,

(*b*) Immersed in a 10 per cent sulfuric acid solution containing a commercial inhibitor,

(*c*) Brushed with a wire brush,

(*d*) Washed in hot water,

(*e*) Dipped in dilute caustic soda solution,

(*f*) Washed in hot running water, and

(*g*) Dried.

After cleaning by this method the interior surfaces of the specimens were clean and bright. The time of immersion in the inhibited acid was generally not more than 1 hr. except for those specimens removed after 10 yr. exposure. The corrosion products on these specimens were quite adherent and sometimes required several hours immersion before they could be completely removed. The alkali dip prevented the slight corrosion and discoloration which would otherwise have occurred during drying. Early in the investigation the efficacy of the cleaning procedure was tested by subjecting uncorroded samples of the various materials to the complete cleaning cycle. The "blank" losses thus obtained were found to be so small that they were disregarded in the final calculations.

The specimens were weighed and the corrosion losses were calculated in grams per square decimeter of interior surface area. The specimens were then sawed longitudinally and the depths of the pits on the corroded surfaces were measured in the following manner:

Early in the test, when the pits were comparatively shallow, a depth micrometer was used for their measurment. In the later stages when the pits were much deeper a micrometer caliper with a pointed spindle was used. The point fitted into the bottom of the pits with the anvil of the caliper in contact with the outer semi-circular surface of the pipe. The thickness of the wall was measured at the bottom of the pit and the pit depth was determined by subtracting this thickness from that of either (*a*) the

---

[5] The number of specimens was determined by the conditions of the experiment from time-to-time and not by a predetermined plan.

TABLE III.—AVERAGE COMPOSITE MINERAL ANALYSES OF WATER (PARTS PER MILLION) WASHINGTON, D. C.

Year ending June	Total Solids	Organic Matter	Silica	Iron	Calcium	Magnesium	Chloride, Cl_2	Sulfate, SO_4	Bicarbonate, HCO_3	Total Alkalinity, $CaCO_3$	pH	Carbon Dioxide	Total Hardness, $CaCO_3$	Non CO_3 Hardness	O_2 Consumed
1936	140	46	1.6	0.03	31	3.1	6.3	31	70	57	7.7	2.0	82	25	0.5
1937	142	46	2.0	0.03	29	3.9	5.8	31	74	56	7.5	2.6	90	34	0.7
1938	128	39	1.9	0.04	28	3.7	5.9	29	64	51	7.7	1.7	83	32	0.7
1939	131	38	2.0	0.06	26	4.1	5.6	30	64	53	7.8	1.8	85	29	0.6
1940	134	39	2.8	0.07	30	3.3	4.7	33	69	57	7.8	1.9	87	28	0.9
1941	130	38	2.2	0.03	26	4.4	6.4	28	68	59	7.9	1.2	84	25	1.2
1942	158	49	2.4	0.03	34	5.2	4.1	36	82	66	7.9	2.0	103	36	1.3
1943	132	21	6.0	0.04	27	5.5	4.1	29	68	55	7.7	2.9	88	31	1.3
1944	143	18	4.9	0.04	31	6.4	6.0	36	80	67	7.6	3.3	101	35	1.8
1945	138	14	4.8	0.04	28	5.8	2.9	35	76	64	7.7	3.7	98	34	1.1
Average	138	35	3.1	0.04	29	4.5	5.2	32	72	59	7.7	2.3	90	31	1.0
Max.	225	70	9.4	0.16	70	10.1	10.1	53	122	100	8.1	8.7	148	48	...
Min.	81	11	0.6	0.01	18	1.2	0.5	20	35	29	7.1	0.3	60	10	...

TABLE IV.—AVERAGE LOSS OF WEIGHT.[a]

Exposure Period, months	Average Loss of Weight, g. per sq. dm.									
	Material A	Material B	Material C	Material D	Material E	Material F	Material G	Material H	Material I	Material J
6	6.1	5.6	6.0	1.5	6.0	6.7	6.6	5.1	5.8	5.0
12	9.1	9.0	10.2	8.1	9.4	9.3	8.9	9.2	9.5	7.9
18	12.4	11.2	—	—[b]	12.5	12.5	10.6	11.0	—[b]	—[b]
24	11.7	11.5	13.7	4.8	12.8	14.1	11.4	11.8	13.9	10.2
30	12.3	13.1	—[b]	—[b]	13.4	13.5	12.1	12.7	—[b]	—[b]
36	13.9	13.3	15.5	10.2	13.8	15.1	14.2	14.0	15.4	12.9
42	17.9	17.7	16.6	9.1	16.2	15.9	17.1	13.5	14.3	15.9
48	16.7	15.7	17.3	11.7	15.2	16.4	15.3	15.8	17.3	15.7
54	15.4	15.1	—[b]	—[b]	17.1	19.2	15.3	16.1	—[b]	—[b]
60	18.3	16.0	24.0	21.0	17.8	18.9	16.7	18.6	19.9	18.4
72	22.2	20.2	16.7	24.2	22.8	20.5	19.3	21.7	22.2	20.0
84	24.1	26.3	24.2	10.7	25.0	21.7	21.3	22.1	28.1	21.3
120	33.2	23.9	—[b]	—[b]	27.4	26.8	26.5	22.4	—[b]	—[b]

[a] Based on results for one to five specimens; see Table II.

[b] A dash indicates that no specimen was exposed for that period.

TABLE V.—AVERAGE DEPTH OF FIVE DEEPEST PITS (mils).[a]

Exposure Period, months	Material A	Material B	Material C	Material D	Material E	Material F	Material G	Material H	Material I	Material J
6	19	19	none	none	18	17	15	14	23	14
12	27	23	13	none	19	17	18	25	34	22
18	25	25	—[b]	—[b]	27	22	30	24	—[b]	—[b]
24	32	42	17	22	31	20	30	27	38	25
30	31	39	—[b]	—[b]	21	24	19	20	—[b]	—[b]
36	38	40	15	24	36	25	30	33	46	25
42	28	33	none	none	22	28	19	28	49	31
48	39	41	17	none	37	27	35	27	48	27
54	39	44	—[b]	—[b]	34	27	35	38	—[b]	—[b]
60	39	44	29	27	39	35	30	31	53	30
72	37	44	22	37	36	40	38	31	54	34
84	39	55	27	51	47	37	45	31	62	30
120	50	55	—[b]	—[b]	46	50	50	39	—[b]	—[b]

[a] Based on results for one to five specimens; see Table II.

[b] A dash indicates that no specimen was exposed for that period.

TABLE VI.—DEPTH OF DEEPEST PITS (mils).[a]

Exposure Period, months	Material A	Material B	Material C	Material D	Material E	Material F	Material G	Material H	Material I	Material J
6	26	29	none	none	27	25	22	24	26	19
12	36	27	16	none	30	25	25	29	43	30
18	40	28	—[b]	—[b]	37	31	40	26	—[b]	—[b]
24	36	51	19	23	35	24	33	31	40	27
30	35	42	—[b]	—[b]	25	29	22	23	—[b]	—[b]
36	55	65	17	28	45	35	39	38	56	32
42	30	42	none	none	24	29	21	32	56	31
48	54	57	30	none	52	45	54	35	54	50
54	40	55	—[b]	—[b]	45	30	55	47	—[b]	—[b]
60	48	64	42	35	48	51	47	39	68	42
72	53	64	34	61	47	64	52	46	72	53
84	51	109	34	61	56	56	55	46	68	46
120	76	70	—[b]	—[b]	54	81	61	74	—[b]	—[b]

[a] Based on results for one to five specimens; see Table II.

[b] A dash indicates that no specimen was exposed for that period.

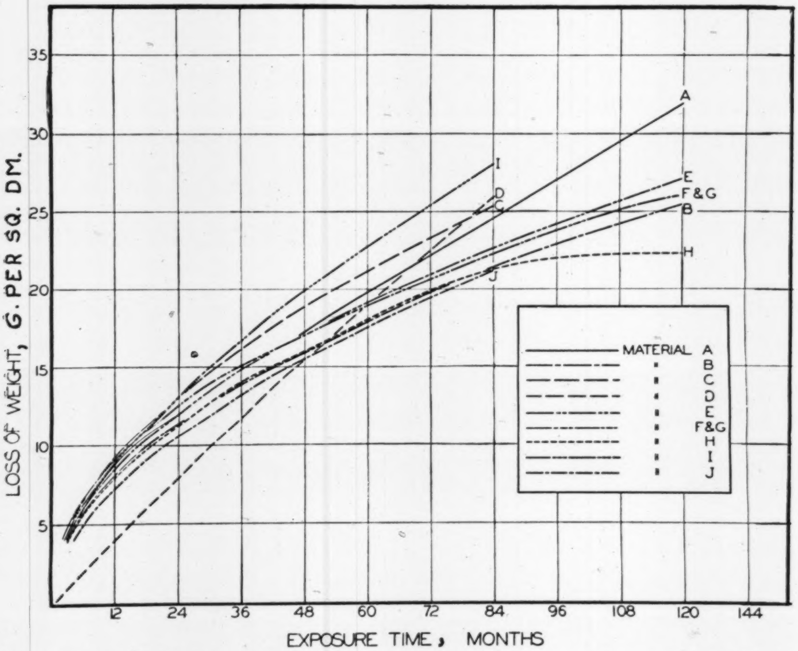


FIG. 4.—Loss of Weight of the Various Materials During the Progress of the Test.

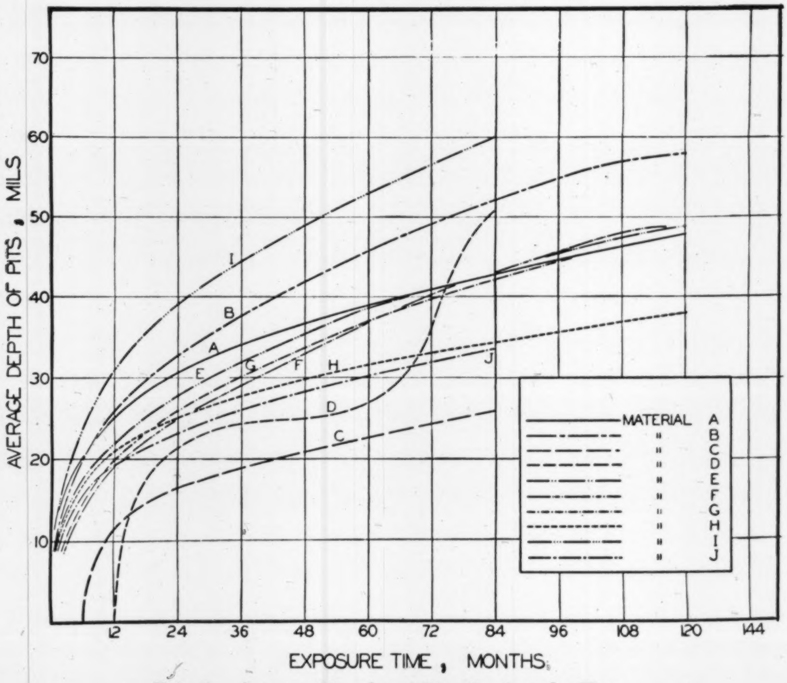


FIG. 5.—Average Depths of Pits During the Test.
The depth is the average of the five deepest pits for each specimen tested.

nearest uncorroded high spot or (*b*) the original wall thickness of unexposed specimens of the same material. Method (*a*) was used in the early stages of the test when considerable portions of the interior surfaces were uncorroded. However, in the later periods it was difficult to find uncorroded areas and it was necessary to resort to method (*b*). Twenty measurements were made at random on each specimen, ten on each half section.

WATER

The water used for the investigation was Washington, D. C., city water taken directly from the supply line. The average composite mineral analyses for each year of the test as obtained from the District of Columbia Filtration Plant are given in Table III. The maximum and minimum values represent the extreme range of single analyses for the entire 10-yr. period.

RESULTS AND DISCUSSION

The corrosion of the interior surfaces of the pipes started with the formation of small localized mounds of rust, the number and size of which increased with time. The rust which formed during the first year or two was lightly adherent and was easily removed. As the corrosion continued, the rust coating became denser, more tightly adherent, and was removed with greater difficulty. Most of the mounds developed into a tubercular form of rust. Many uncorroded areas were found on the surfaces of most of the specimens during the first several years of testing, but few were found on specimens exposed for more than 5 yr. The pH-alkalinity relationship of the water was such that small amounts of calcium carbonate were deposited with the rust, probably increasing the protective effects of the rust coating.

The results of corrosion tests usually show considerable scatter among the individual specimens, and the results of this test are no exception. A rather definite trend was established, however, early in the test and continued throughout its life. Materials which showed the highest losses of weight or depths of pits in the early stages generally maintained this trend throughout.

The average loss of weight for each of the materials after each exposure period is given in Table IV, the average depth of the five deepest pits in Table V, and the depths of the deepest pits in Table VI. The variation of the loss of weight and average depths of pits with time are shown as curves plotted by a least squares method in Figs. 4 and 5, respectively. It was not possible to plot similar curves for the depths of the deepest pits because of inconsistent results obtained during the various test periods.

The loss of weight of all of the wrought materials and the centrifugally cast iron pipe C was relatively rapid during the early stages of the test, after which the rate decreased gradually and became more or less constant. The sand-cast iron pipe D behaved in a somewhat erratic manner and the best curve which could be drawn was a straight line. At the end of seven years, however, this material had experienced about the same weight loss as that of the centrifugally cast iron.

Since there were not enough specimens to test all of the materials for 10 yr., comparisons of the results cannot be extended beyond the 7-yr. period. At this point the specimens fell into three groups, materials B, E, F, G, H, and J having the lowest corrosion losses, materials A, D, and C with somewhat higher losses, and material I, which had the highest loss. This grouping is, of course, sensitive to the length of exposure at which the comparisons are made.

For those specimens tested for 10 yr. the trend was approximately the same as for 7 yr. The only exception was a decrease in the corrosion rate of material H.

The average depths of pits at the end of 7 yr., Table IV and Fig. 5, showed considerably more difference than the average losses of weight. The results indicated that the materials could be divided into three groups, materials C, J, and H having the shallowest pits; materials A, E, G, and F intermediate pit depths; and materials D, B, and I the deepest pits. There was some scatter in the results obtained from individual specimens of each material, due probably to localized concentration of the corrosion attack at certain points by selective action.

The shallowest pits occurred in the centrifugally cast iron, while the sand-cast iron had pits approximately twice as deep. Since the loss of weight was about the same for each material, the results indicate that the corrosion of the centrifugally cast iron was general, with little localized deep pitting; that of the sand-cast iron was largely by deep pitting. The condition of the casting skin of these materials was probably largely responsible for this difference in corrosion characteristics.

Wrought iron B, which had a relatively low loss of weight, had deep pits, indicating that the corrosion attack on this material was localized.

Steel I, which had the greatest weight loss, likewise had the deepest pits. The general grouping of the other materials was very similar both as regards the loss of weight and the depth of pits.

The depths of the deepest pits, Table VI, showed considerable variation even among specimens exposed for the same length of time. While the general trend was to show deeper pits with increased exposure time, there are numerous exceptions, particularly in the case of material A. One of the pits in a specimen exposed for 36 months was 65 mils deep. This depth was not approached again for this material until a specimen was examined after 60 months of exposure. After 84 months of exposure, one of the specimens contained a pit 109 mils deep, whereas the deepest pit measured after 120 months was 70 mils. This apparent wide scatter in results is due probably to either or both of two causes—(1) inadequacy of measurements, since but twenty pits were measured on each specimen, and (2) the localization of attack in favorable areas in certain specimens.

The average depths of the five deepest pits showed considerably less variation than the deepest pit measured for each material. This probably arises from the fact that the average of the five deepest pits in twenty measurements is subject to less variation than is the single measurement of the deepest individual pit (due to sampling material that is not perfectly homogeneous). Hence, the average depths of the five deepest pits out of twenty provide a more precise estimate of that parameter of pitting tendency than is obtained from the depth of the deepest pit.

Failure of any piping material in actual service usually results from perforation of the wall by pitting, with resultant leakage. It might be expected that the depth of the deepest pit would control the time required for a pipe to pit completely through, but no evidence was found to indicate that the deepest pit at any intermediate stage of the test would be necessarily the first to perforate. On the other hand, some evidence was obtained that pitting might be inhibited at times by the deposition of chemical compounds, or by the formation of corrosion products in the bottoms of the pits, thus preventing or slowing up subsequent attacks. Excluding the two cast irons, which had greater wall thicknesses than the wrought materials, the average depths of pits after 7 yr. of testing represented penetrations of from 20 to 40 per cent of the wall thickness, whereas after 10 yr., the penetrations were from 25 to 36 per cent. It should be noted that one material, I, had deeper average penetration after its longest exposure period of 7 yr. than any of the materials which were tested for 10 yr. If the depths of the deepest pits are used as criteria, after 7 yr. of testing, again excluding the cast irons, the penetration of corrosion into the various materials ranged between 30 and 70 per cent, while those tested for 10 yr. ranged from 36 to 52 per cent. Under these conditions a rough estimate of the minimum life of uncoated pipes of this type and for this kind of service would be about 15 yr.

Comparison of the average depths of pitting obtained in these tests with those obtained in other tests still under way at the National Bureau of Standards on actual service lines using the same water indicates that approximately 50 per cent greater corrosion occurred in the laboratory tests than in the service lines. This is probably due to the continuous flow of the water in the laboratory tests as compared with intermittent flow in the service lines. In the laboratory tests, the corrosion products are probably removed more rapidly than in the service lines; in the latter the accumulation of corrosion products probably slows down the corrosion process to some extent.

At the end of the 10-yr. test, all of the samples contained corrosion products which occupied approximately one-fourth of the cross-sectional area of the pipes. Their hydraulic efficiency in regard to carrying water was undoubtedly very low under these conditions.

SUMMARY

The results of the tests indicated that there was no great difference in the rates of corrosion of most of the wrought materials, when measured either by loss of weight or by depth of pitting. A low-alloy steel had the highest corrosion rate, while a copper-molybdenum ingot-iron and a nickel-bearing wrought iron had the lowest rates. Other low-alloy materials, wrought irons, ingot iron and ordinary medium carbon steel pipes corroded at intermediate rates. Since all of the materials were tested in the uncoated condition, the pipes contained considerable corrosion products at the end of the test, which decreased their hydraulic efficiencies to a considerable extent. Estimated roughly from these tests, the minimum length of life of these bare materials when subjected to continuous flow of Washington, D. C. water would be about 15 yr.

These tests, conducted under controlled laboratory conditions, indicated corrosion rates about twice as great as those in service lines at the National Bureau of Standards, on the basis of tests not yet completed.

Acknowledgment:

The authors wish to acknowledge the valuable assistance rendered at various times by Mr. H. Babb, Miss Ellen Connelly, and Mrs. Phyllis C. Deming.

AN INVESTIGATION OF THE POSSIBILITIES OF ORGANIC COATINGS FOR THE PREVENTION OF PREMATURE CORROSION-FATIGUE FAILURES IN STEEL*

By Robert C. McMaster[1]

Synopsis

Repeated stressing of steels exposed to corrosive brines often causes premature corrosion-fatigue failures. A few readily available organic coatings were investigated as possible means of protection. The results of the laboratory tests were confirmed by field experience in the oil-well drilling industry.

Room-temperature R. R. Moore rotating-beam fatigue tests were made of bare specimens of S.A.E. 1045 steel operating in air and in a corrosive brine containing 7 per cent total salts. Single specimens with intact and with scratched organic coatings were tested in the brine.

The hot-rolled and normalized steel had a static ultimate tensile strength of 107,000 psi. The yield strength was 62,500 psi. The endurance limit in air under reversed bending stresses was about 48,000 psi. The corrodent had little effect upon the 1750 cycle per minute operating life of bare steel, at stresses above 48,000 psi. Below this stress level, the corrosion-fatigue life of bare specimens increased about 10 times for each 20,000 psi. reduction in the applied stress.

Organic coatings significantly lengthened the corrosion-fatigue life at stresses below 48,000 psi. in these tests. A typical four-coat system increased the operating life at 40,000 psi. maximum stress from 430,000 cycles (bare) to more than 64,000,000 cycles (coated). This coating contained 60 per cent alkyd-type and 40 per cent urea-formaldehyde type resin solids as a binder. It contained a protective pigment in the primer coats, and was baked at 250 F. after each coat. A five-coat system of air-drying materials increased the operating life under the same test conditions to more than 37,000,000 cycles.

Several additional complete organic coating systems were prepared. These specimens have not yet been tested. There is little hope of causing these coatings to fail under the standard test conditions (40,000 psi. maximum reversed stress, 1750 cycles per minute) in periods shorter than several months.

From these tests it appeared possible that intact organic coatings might provide great help in preventing premature corrosion-fatigue failures of steels in industrial equipment subjected to corrosive environments. However, *after failure* of the organic coatings (simulated in the tests by scratches cut through the coatings), the remaining operating lifetime was found to be about the same duration as that of new, bare specimens.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Assistant Research Supervisor, Battelle Memorial Institute, Columbus, Ohio.

Many industrial processes subject steels to repeated stresses in the presence of corrosive brine, with consequent failures resulting from corrosion-fatigue. Such failures typically develop from corrosion pitting which produces stress concentrations. Fatigue cracks propagate from the points of stress concentration at a rate which is often augmented by the wedging action of the products of corrosion which form in the cracks.[2] The operating life of the steel under corrosion-fatigue conditions may be greatly decreased by the combined effects of the chemical attack and repeated stresses. Dangerous premature failures often bring attention to these conditions only after extensive corrosion-fatigue damage has occurred to large installations, when correction may be difficult or extremely expensive.

Particularly severe corrosion-fatigue damage problems are encountered in the oil-well drilling industry in the Permian Basin region of west Texas (**1**).[3] Brine is used there as a drilling fluid while drilling through the salt beds. Similar difficulties are encountered in off-shore drilling operations where sea water is used as a drilling fluid. As early as 1928, Speller *et al.* (**2**) proposed that rotary drill pipe be protected from corrosion-fatigue damage by suitable protective coatings. However, bare drill pipe was used almost exclusively up to 1945. The increasing number of failures of drill strings in the hole, and the requirements of deeper drilling, forced the drilling industry to reconsider coatings as a possible means of preventing premature corrosion-fatigue failures in steel drill pipe. Laboratory and field investigations were then made to evaluate possible methods of prolonging the operating life of drill pipe (**3**, **4**, **5**, **6**, **7**).

Several previous investigations had been made of the possibilities of applying metallic coatings to prevent premature corrosion-fatigue failures in steel. Swanger and France (**8**) found that hot-dipped galvanized zinc coatings reduced the (pure) fatigue properties of carbon steels tested in air in the absence of corrosion. Electrodeposited zinc coatings resulted in endurance limits (in air) equal to or greater than those of bare specimens. Watt (**9**) explained these results by his observation that hot-dip zinc coatings developed, under reversed stressing, closely spaced hair-line cracks perpendicular to and terminating at the surface of the steel. Electroplated zinc coatings showed no evidence of such cracks after similar treatment. Kenyon (**10**) made corrosion-fatigue tests of electroplated steel wires. He found that plating with a more noble metal, such as copper, might contribute to the embrittlement of steel wire. Numerous fatigue cracks resulted from galvanic action at points of imperfection in the plating. The possibilities of both electroplated zinc and cadmium coatings, and of bakelite phenolic varnish coatings, were investigated by Dolan and Benninger (**11**), as a means of improving the corrosion-fatigue life of S.A.E. 4130 and other steels in fresh tap water. Tests were made with one heat-hardenable and two air-hardenable bakelite varnishes. They concluded that uniformly adhering, impervious coatings might offer an effective means of protecting a steel member from localized corrosive attack, if abrasion were not likely to occur in service.

Modern organic coating materials and techniques offer great promise in the field of protection of steel from corrosive industrial processes. Specific operating conditions may be met by coating systems providing good adherence and chemical inhibitors in the primer layers, and abrasion resistance, hardness, flexibility, alkali resistance, and resistance to salt solutions or other specific chemicals, in the outer layers. Extensive commercial services which supply materials or apply coatings are now available. Techniques have been developed which provide adequate and reliable coverage even in the case of the inside surface of long pipes and tubes such as are commonly used in the oil industry.

---

[2] Similar wedging action by viscous coating fluids has been reported by L, Ferguson and G. Bouton in "The Effect of a Coating of Polybutene on the Fatigue Properties of Lead Alloys," Symposium on Stress-Corrosion Cracking of Metals, Am. Soc. Testing Mats., pp. 473–484 (1944).

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 644.

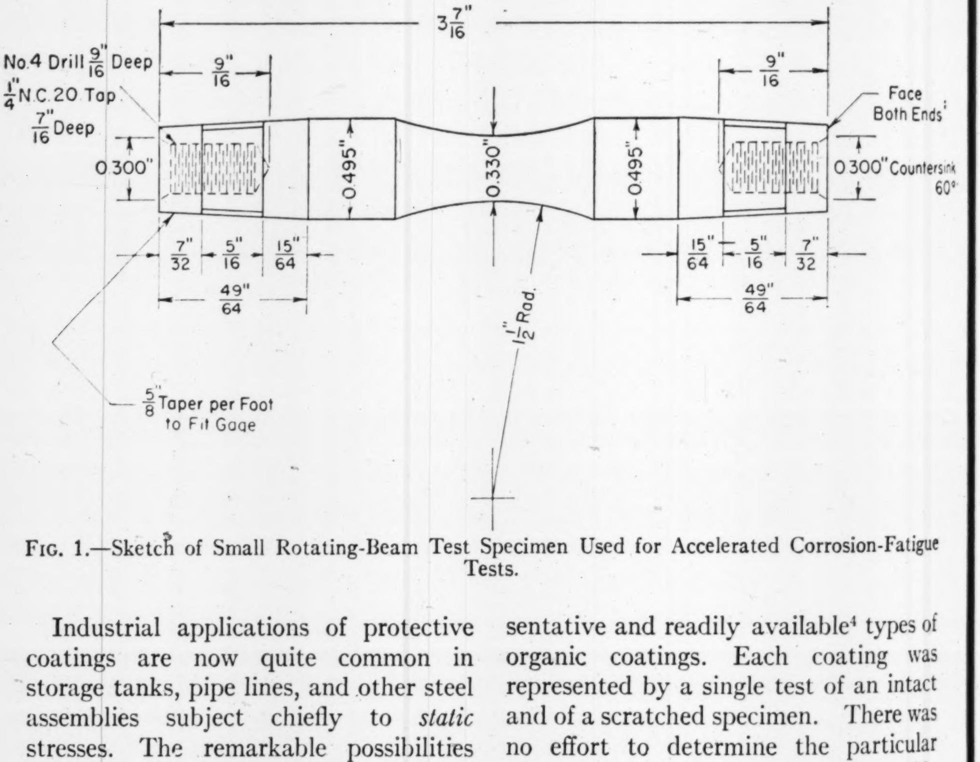


Fig. 1.—Sketch of Small Rotating-Beam Test Specimen Used for Accelerated Corrosion-Fatigue Tests.

Industrial applications of protective coatings are now quite common in storage tanks, pipe lines, and other steel assemblies subject chiefly to *static* stresses. The remarkable possibilities of organic coatings for protecting steel which is subjected to both corrosion and *repeated* stresses have not been so widely recognized or applied in industry. Organic coatings have recently been applied to the inner surface of oil-well drill pipe. This steel is subjected to extreme conditions of corrosion and repeated stressing, and to frequent handling and mechanical abuse. Coated drill pipe is a dramatic example of what may be done. The control of corrosion-fatigue in many industrial applications may be expected to offer less difficulty.

Laboratory Tests

A series of laboratory tests was undertaken to determine the magnitude of the possible improvements in operating life of steel repeatedly stressed in the presence of a corrosive brine, which might be attained with a few representative and readily available[4] types of organic coatings. Each coating was represented by a single test of an intact and of a scratched specimen. There was no effort to determine the particular *types* of coatings most suited for specific applications. The relative merits of the different coatings might have been changed by averaging the results of additional tests or by comparisons at other stresses.

In these tests, the steel was first subjected to fatigue tests in the absence of corrosion, to provide reference data corresponding to complete protection from corrosion. Next, the steel was subjected to fatigue tests under severe corrosion conditions, with no protection. Succeeding tests were made with single-layer organic coatings without pigments, to compare the properties of resins used as binders and surface coats. Finally, complete coating systems were tested, both as intact coatings and with scratches purposely cut through the coatings prior to testing.

---

[4] In 1945–1946.

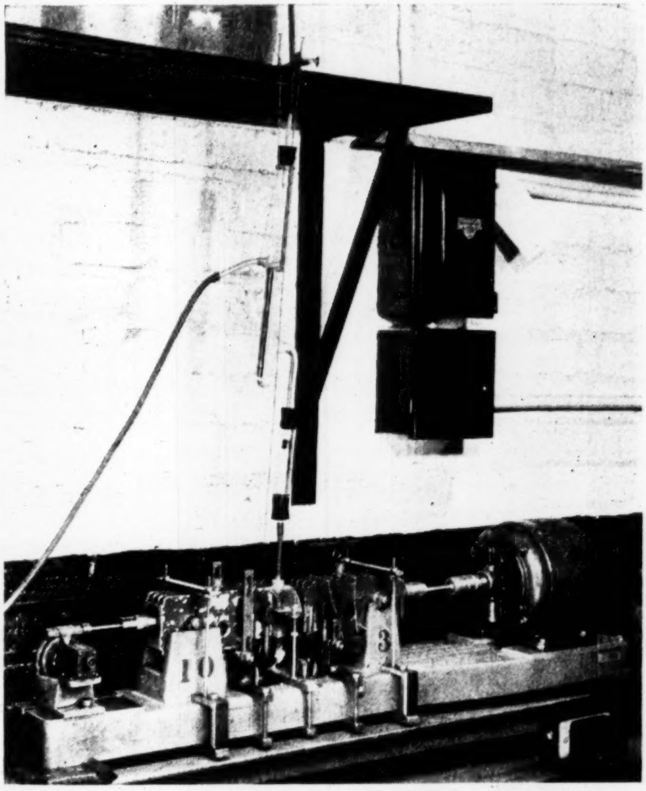


Fig. 2.—Equipment for Accelerated Corrosion-Fatigue Testing Showing R. R. Moore Rotating-Beam Fatigue Testing Machine, Equipped with Krouse Plastic Corrosion Test Cell and System for Circulating Corrosive Brine over Test Specimen.

Testing Equipment and Methods

The corrosion-fatigue tests were made at room temperature with small unnotched steel specimens (Fig. 1). These were repeatedly stressed at 1750 rpm. on conventional R. R. Moore rotating-beam fatigue testing machines such as that shown in Fig. 2. The corrodent was dropped upon the rotating specimen at its section of minimum diameter. The rate of application was controlled by a capillary tube under a fixed head or pressure. An excess of corrodent was circulated past the secondary reservoir above the capillary to maintain the constant head (note arrangement of glass feed tubes in Fig. 2). A plastic "Krouse" cell confined the corrodent to the portion of the specimen under test. Rubber slingers prevented the flow of corrodent along the specimen to the bearings of the testing machine. Preliminary tests indicated that variations in the rate of application of corrodent, so long as at least one drop per second fell upon the specimen, did not significantly influence the fatigue life of the specimen. Provision was made so that the driving motor and the flow of corrodent would be stopped when the specimen failed. The total number of revolutions was recorded on a suitable counter.

The corrodent selected as standard for these tests contained:

	Parts per million	per cent
Sodium chloride	66 000	6.6
Magnesium chloride	1 000	0.1
Calcium chloride	3 000	0.3
Distilled water	Balance	Balance

This corrodent corresponded to standard A.P.I. corrosive brine, save that no hydrogen sulfide was introduced. Oxygen was present, since the specimens were tested in air. No additional oxygen was purposely introduced into the corrodent. The brines were made up as fresh solutions for each test. The brine which had passed over the test specimens was drained off and not used again.

Preliminary tests indicated that more dilute or more concentrated solutions of the corrodent were less effective in reducing operating life than the 7 per cent total salt content selected for the standard test condition.

The rotating-beam test specimens were machined from 0.505-in. diameter S.A.E. 1045 hot-rolled and normalized steel rod. The dimensions are shown in Fig. 1. The static test properties of the steel were:

Tensile strength, psi.	106 500	to	108 000
Yield strength, psi.	62 400	to	62 500
Elongation in 2 in., per cent	24.5	to	25.4
Reduction, in area, per cent	48.9		

BASIC STRESS-CYCLE CURVE (*S-N*) DATA FOR BARE TEST SPECIMENS

Preliminary tests were made of bare test specimens operating in air without application of corrodent, to provide a basic stress-cycle curve for the steel in the absence of corrosion (Fig. 3, upper curve). From this stress-cycle (*S-N*) curve, it is apparent that the operating life at a high maximum stress of 70,000 psi. was very short, only 20,000 cycles or load applications. When the maximum stress was reduced to 50,000 psi., the life increased to at least 175,000 cycles. At stresses below the endurance limit of 48,000 psi., the specimens did not fail, even after as many as 50,000,000 cycles of load.

Typical fatigue fractures obtained in these tests are shown in Fig. 4. These were normal "pure fatigue" failures which propagated, in most cases, from a single initial crack or fatigue nucleus, in the reduced section where maximum stress was applied.

A second group of bare specimens was subjected to fatigue tests in the Krouse cell, with the standard corrodent applied by dropping it upon the specimen at a rate of about one drop each second. This test condition produced accelerated corrosion and provided stress-cycle data for completely unprotected specimens under severe corrosion conditions (Fig. 3, lower curve).

The curves shown in Fig. 3, for uncorroded and corroded specimens, indicate that at this speed of testing (1750 rpm.) and at stresses above 48,000 psi., there was only a slight reduction in lifetimes due to corrosion. This reduction was small in magnitude in comparison with the width of the scatter bands of the curves. No reliable evaluation of methods of reducing corrosion could be made from specimens tested at stress levels above 48,000 psi.

Below 48,000 psi., however, the difference between the fatigue lifetimes of corroded and uncorroded specimens was very great, since the uncorroded specimens did not fail at stresses below the endurance limit. On the other hand, the corroded specimens failed at finite lifetimes at any repeated stress level, no

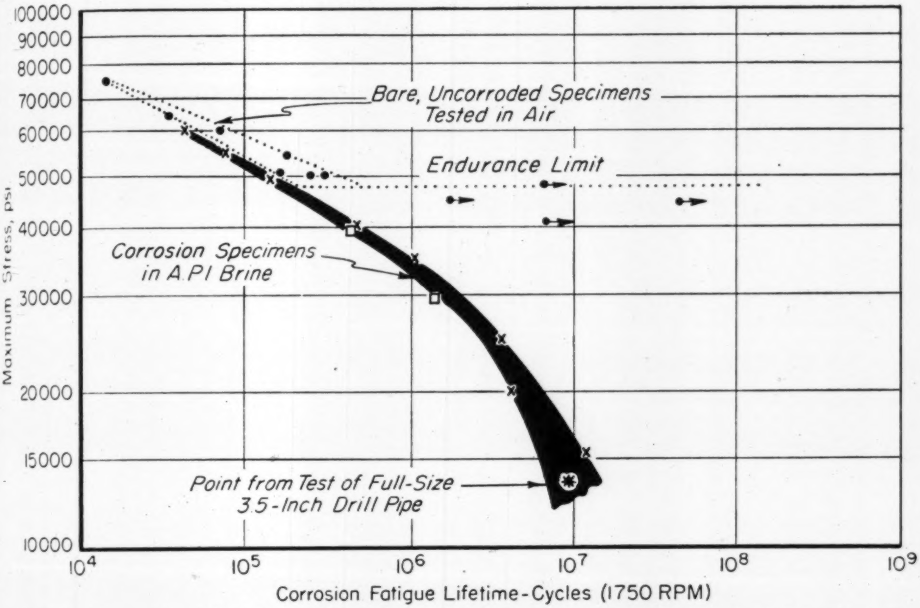
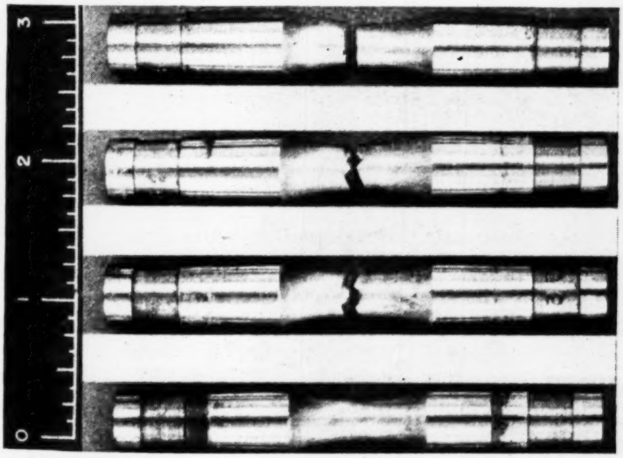


FIG. 3.—Stress-Cycle Data for Bare Specimens Tested in Air and in the Standard Corrodent.

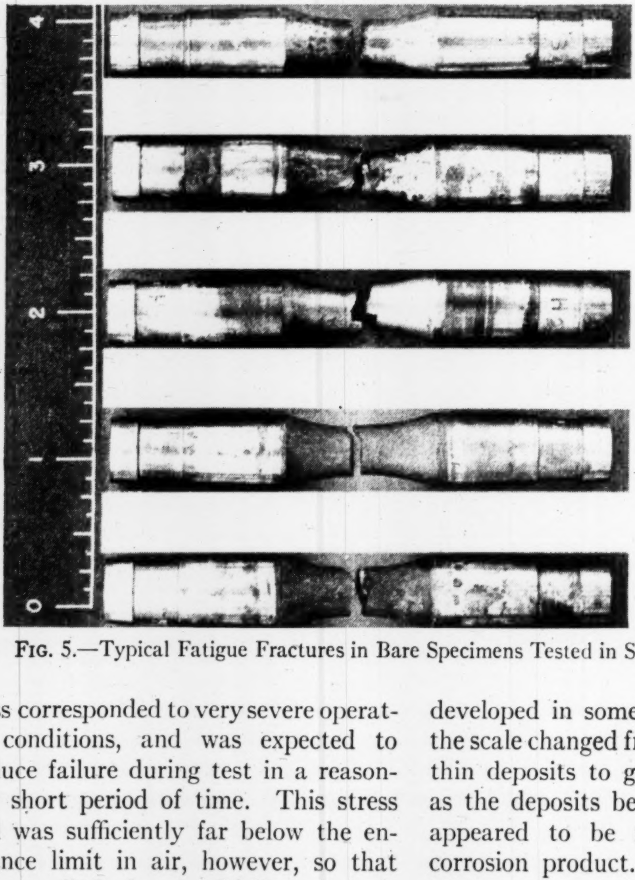


Maximum Stress, psi.	Fatigue Lifetime, cycles
75,000	15,000
55,000	92,100
50,000	299,300
47,500	7,010,000+ (no failure)

FIG. 4.—Typical Fatigue Fractures in Bare, Uncorroded Specimens Tested in Air.

matter how low. In this region, the lower the maximum stress, the longer the fatigue lifetime in the presence of corrosion. The scatter of test points was greater at low stresses and long lifetimes, a natural consequence of the irregular nature of corrosive attack on steel.

From these data, a maximum, fully reversed stress of 40,000 psi. was selected for further comparative tests. This stress corresponded to very severe operating conditions, and was expected to produce failure during test in a reasonably short period of time. This stress level was sufficiently far below the endurance limit in air, however, so that there was little danger of failure through pure fatigue, in unnotched specimens which were completely protected from corrosion. The normal fatigue lifetime of specimens subjected to the standard corrodent at 40,000 psi. maximum stress was between 419,000 and 435,000 cycles as a typical value.



Maximum Stress, psi.	Fatigue Lifetime, cycles
60,000	42,900
40,000	435,100
40,000	399,400
(This specimen was tested fully immersed in standard corrodent in A.P.I. cell.)	
25,000	3,631,300
15,000	11,427,200

Fig. 5.—Typical Fatigue Fractures in Bare Specimens Tested in Standard Corrodent.

Typical fractures resulting from simultaneous corrosion and fatigue loading in these tests are shown in Fig. 5. All failures occurred in the reduced section of the test specimens. The specimens appeared to be more severely corroded as the periods of exposure to the brine increased, with heavy scale layers being developed in some cases. The color of the scale changed from light orange in the thin deposits to green, blue, and black as the deposits became thicker. These appeared to be normal forms of the corrosion product. The inner layer of black rust had the appearance of hydrated ferrous oxide, which is characteristic of corrosion where the air supply is restricted by the outer layers of the deposits. The initial rusting of clean steel specimens and the outer layers of

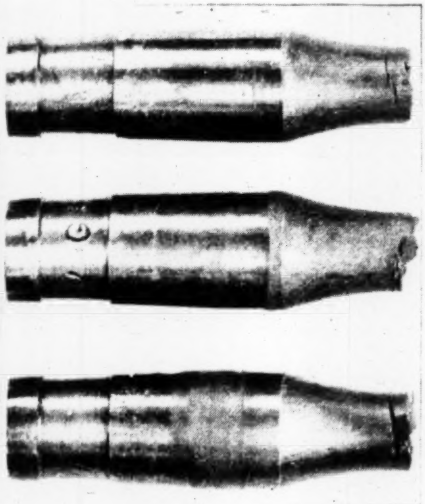


Fig. 6.—Multiple Fatigue Cracks in Bare Specimens Tested in the Standard Corrodent (After Electrochemical Cleaning).

rust on thickly coated specimens resembled hydrated ferric oxide.

After electrochemical cleaning, the surfaces of the corroded specimens had the appearance shown in Fig. 6. The specimens which had short lives showed little damage to the surface and few pits. The specimens which had been under attack from brine for longer periods had more general corrosion and large area pitting. Some of the specimens showed evidence of the formation of secondary fatigue cracks in addition to the crack which developed into a complete fracture. This condition of multiple fatigue cracks, starting from many separate corrosion pits, is quite common in steel operated under cor-

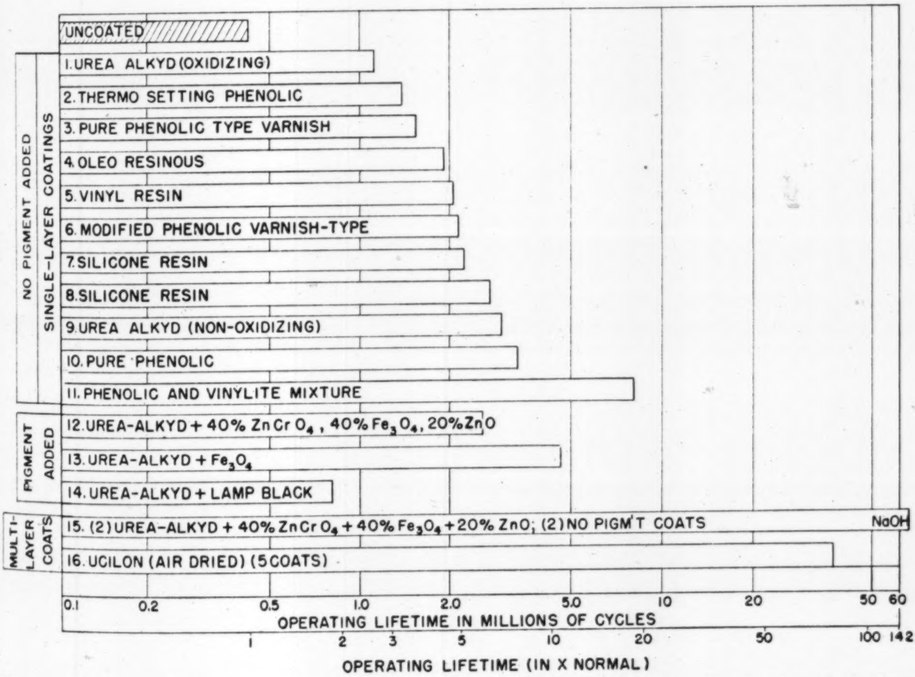


Fig. 7.—Influence of Intact Coatings on the Operating Lifetime of S.A.E. 1045 Steel in Corrosive Brine.

rosion-fatigue conditions. Such multiple cracks are very rare, however, in pure fatigue failures in the absence of corrosion effects.

Two specimens were subjected to fatigue tests while fully immersed in the standard corrodent in an A.P.I. cell (identified by open squares in Fig. 3). The test points fell on the lower edge of the scatter band of points obtained with the Krouse cell, in which corrodent was dropped upon the specimens in the presence of air. The differences in lifetime were so small as to be negligible in fatigue testing. Succeeding tests were therefore all made in the Krouse cell.

Comparative Tests of Single-Layer Organic Coatings Applied Without Pigments

A series of test specimens was prepared with clear, unpigmented organic coatings. These were formulated from quickly available representative resins and other raw materials. Only one coat was applied to each specimen. These tests were expected to indicate, in a reasonably short period of time, the relative performance of the coating materials under repeated reversed strain in the presence of corrosive brine. Probably similar handling of equivalent raw materials from other sources would have given comparable results.

Each specimen was then tested in the standard corrodent at 40,000 psi. maximum stress, fully reversed. The results of the fatigue tests are summarized in Table I. The relative performances of the various coating materials are indicated by the bar charts of Fig. 7.

Coating No. 11, a mixture of phenolic and Vinylite resins, showed unusually good performance. It increased the operating life of specimens in the standard corrodent to about 18 times normal life at 40,000 psi. maximum stress. Other coating materials, applied in single coats without pigments, increased the operating lifetime from 1.8 to 7.8 times normal.

These tests indicated that purely mechanical protection of the surface by means of unpigmented coatings might increase the operating life significantly. Since the completion of these tests, new resins have been announced which offer promise of further improvements in performance in this type of application.

Comparative Tests on an Organic Coating Material with Pigments Which Have Been Classified as Inhibitive, Indeterminate, or Stimulative to Corrosion

Six specimens were coated with one layer of a non-oxidizing, urea-alkyd type of coating in which the pigment varied from inhibitive through indeterminate to supposedly stimulative materials (as classified by Jordan and Whitby (12)) to determine the influence of pigment protection upon the operating life of coated specimens. The properties of the coatings and pigments and the test results are listed in Tables II and IV. The inhibitive pigment contained zinc chromate, zinc oxide, and iron oxide. The indeterminate pigment contained only iron oxide. The stimulative pigment was carbon (lampblack). One specimen of each type was scratched severely before testing. The remaining specimen of each type was tested with the coating intact.

The iron oxide pigment, classified as indeterminate, apparently gave better protection in this limited test than did the inhibitive pigment, in which the zinc constituents were expected to inhibit corrosion. The lampblack, which was classified as stimulative to corrosion, lowered the operating lifetime significantly below that obtained with other pigments or with no pigments at all (Fig. 7).

TABLE I.—DESCRIPTION AND PROPERTIES OF UNPIGMENTED ORGANIC COATINGS USED IN CORROSION FATIGUE TESTS AT 40,000 Psi. MAXIMUM STRESS.

Specimen	Coating Type	Coating Constituents, per cent	Method of Application	Baking Time, min.	Baking Temperature, deg. Fahr.	Film Thickness,[c] mils	Hardness[a]	Abrasion Resistance,[b] revolutions per mil thickness	Abrasion Loss[b]	General Appearance	Corrosion-Fatigue Lifetime, cycles
No. 1.....	Urea-alkyd (oxidizing)	80% alkyd-type resin solids (Duraplex A-25) 20% urea modified melamine-type resin solids (Uformite MU-56)	Spraying	25	300	1.1	16	1540	0.02 g. after 1000 revolutions	Smooth, clear film	1 107 800
No. 2.....	**Thermosetting** phenolic type	100% oil-modified phenolic resin solids (Amberol HH-1)	Spraying	30	350	1.3	24	166	0.015 g. after 200 revolutions	Pits formed in film when baked	1 374 700
No. 3.....	Commercial, pure phenolic-type varnish	28% (minimum) pure phenolic resin 45% (minimum) oil-tung, chinawood, raw 2% (minimum) castor oil 25% (minimum) linseed oil, raw	Spraying	30	250	0.7	20	100	0.015 g. after 100 revolutions	Film wrinkled when baked	1 535 500
No. 4.....	Oleo-resinous	58.8% linseed oil, varnish type 4.5% pentarerythritol 36.7% wood resin	Spraying	30	250	0.7	28	170	0.012 g. after 100 revolutions	Smooth, clear film	1 940 000
No. 5.....	Vinyl-resin (plasticized)	65% vinyl resin solids 38.5% Vinylite VYLF 61.5% Vinylite VYHH 15% dioctyl phthalate 20% methacrylate-type resin (Acryloid B-72)	Spraying	20	350	1.5	22	1000	0.012 g. after 1000 revolutions	Blackened when baked, had poor adhesion	2 048 600
No. 6.....	Modified phenolic **varnish type**	34% modified phenolic resin (Reichhold Chemical Company, Beckacite No. 1123) 53% china wood oil 13% linseed oil	Spraying	(dried in air 4 days)		1.5	not measured	264	0.014 g. after 200 revolutions	Slight wrinkling	2 134 200
No. 7.....	Silicone resin type	83% Dow Corning silicone resin DC-801 17% Dow Corning silicone resin DC-803	Spraying	60 and 60	250 300	1.2	not measured	212	0.060 g. after 250 revolutions	Smooth, clear film	2 204 200
No. 8.....	Silicone resin type	54% Dow Corning resin DC-801 46% Dow Corning resin DC-803	Spraying	40 and 60	250 300	1.2	not measured	303	0.05 g. after 300 revolutions	Smooth, clear film	2 695 000
No. 9.....	Urea-alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea aldehyde-type resin solids (Beckamine P-138)	Spraying	45	300	0.9	14	748	0.05 g. after 1000 revolutions	Smooth, clear coating	2 937 700
No. 10.....	Pure phenolic varnish type	28% pure phenolic (Bakelite BR-254) 23.7% raw linseed oil 46% china wood oil 2.3% castor oil	Spraying	(dried in air 4 days)		1.2	not measured	350	0.02 g. after 200 revolutions	Slight wrinkling	3 350 200
No. 11.....	Phenolic and vinylite mixture	80% phenolic resin (Bakelite XV-16607) 20% polyvinyl butyral (Vinylite XYHL)	Spraying	60 and 30	230 300	0.5	not measured	373	0.02 g. in 500 revolutions	Orange peel	8 037 900

[a] Measured on Sward Hardness Rocker—standard 100 on plate glass.
[b] Measured on Taber Abraser, 1000 load, CS-15 wheel, on 16-gauge panels coated and baked simultaneously with the coated test specimens.
[c] Measured by micrometer.

The single-layer specimens whose coatings had been scratched before testing all failed at relatively short lifetimes, varying from 70 per cent to 136 per cent of the lifetime of bare specimens tested under similar conditions (Fig. 8). These data tended to indicate that organic coatings provide effective protection only while they remain intact and impervious to the corrodent, regardless of pigment. Upon failure of the coating through cracking or scratching, the operating lifetime which remains could not ordinarily be expected to be appreciably longer than that of new bare specimens. For this reason, it appeared probable that properties such as hardness, flexibility, wear resistance, alkali and salt solution resistance inherent in the coating material may have more significance than the inhibitive properties of the pigments.

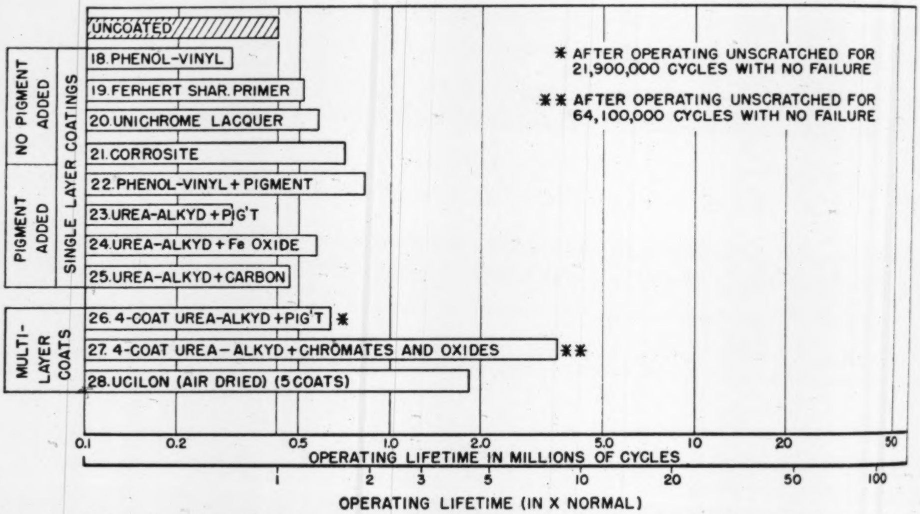


Fig. 8.—Influence of Scratched Coatings on the Operating Lifetime of S.A.E. 1045 Steel in Corrosive Brine.

Comparative Tests of Complete Coating Systems

Commercial organic coating systems commonly contain multiple coats. The individual coats often have different functions. The inner coats may act as primers, insuring good adhesion and providing protective pigments. The outer coats often supply the necessary hardness or wear and abrasion resistance, and resist chemical attack. Two such complete coating systems were tested under the same conditions of corrosion and fatigue loading as used for specimens previously reported, with the results listed in Table III.

The improvements in the operating lifetime of test specimens with these complete coating systems were remarkable. One coated specimen ran for more than 150 times the normal operating lifetime of bare specimens and showed no signs of damage to the coating when the tests were discontinued. Specimen 15, with four coats of urea-alkyd type resin containing an iron oxide-zinc chromate pigment in the two primer layers, had run more than 50 times the normal lifetime when a bearing failed in the testing machine.

TABLE II.—DESCRIPTION AND PROPERTIES OF INTACT, PIGMENTED, SINGLE-LAYER, ORGANIC COATINGS USED IN CORROSION-FATIGUE TESTS.

Specimen	Coating Type	Binder Constituents, per cent	Pigment Constituents, per cent	Ratio of Pigment to Binder by Volume	Method of Application	Baking Time, min.	Baking Temperature, deg. Fahr.	Film Thickness, mils	General Appearance	Corrosion-Fatigue Lifetime, cycles
No. 12...	Urea-alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	40% zinc yellow (zinc chromate) 40% iron oxide (Canox Superfine) 20% zinc oxide (ZZZ-22)	40:60	Spraying	30	300	1.4	Very slight pitting	2 512 000
No. 13...	Urea alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	100% iron oxide (Canox Superfine)	40:60	Spraying	30	300	1.1	Good	4 550 000
No. 14...	Urea alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	100% carbon (Black Knight Velvet lampblack)	40:60	Spraying	30	300	1.0	Good	808 700

TABLE III.—DESCRIPTION AND PROPERTIES OF COMPLETE, MULTILAYER ORGANIC COATING SYSTEMS USED IN CORROSION-FATIGUE TESTS AT 40,000 Psi. MAXIMUM REVERSED STRESS.

Specimen	Type of Coating	Binder Constituents, per cent	Pigment Constituents, per cent	Pigment to Binder Ratio	Method of Application	Baking or Air-Drying Procedures	Corrosion-Fatigue Lifetime, cycles
No. 15...	Primer (two coats)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	40% zinc chromate 40% iron oxide 20% zinc oxide	40:60	Spraying	15 min. forced drying or baking at 250 F. after each coat. System baked 45 min. at 300 F. after last coat.	21 895 000 (15*a*)[a] 64 136 800 (15*b*)[a]
	Finish (two coats)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	Nonpigmented	...			
No. 16...	Primer (two coats)	Ucilon Primer 405-P	Nonpigmented	...	Spraying	5 min. drying between primer coats and 30 min. before finish coats applied. 30 min. drying between finish coats. System dried 6 days in air before testing.	37 780 000
	Finish (three coats)	Ucilon White	Nonpigmented	...			

[a] (15*a*) = Specimen No. 15*a*—Test discontinued when bearing failed.
(15*b*) = Specimen No. 15*b*—51,256,900 cycles in standard corrodent and 12,879,900 additional cycles in 7 per cent salt solution containing 2 per cent NaOH.

TABLE IV.—DESCRIPTION AND PROPERTIES OF COATINGS USED IN CORROSION-FATIGUE TESTS OF SPECIMENS SCRATCHED AROUND THEIR SMALLEST DIAMETER.

Specimen	Coating Type	Binder Constituents, per cent	Pigment Constituents, per cent	Ratio of Pigment to Binder by Volume	Method of Application	Baking and Air-Drying Procedures	Film Thickness, mils	General Appearance	Corrosion-Fatigue Lifetime, cycles
No. 18......	Phenolic and vinyl mixture	80% phenolic resin (Bakelite XV-16607) 20% vinyl resin (Vinylite XYHL)	100% iron oxide (Canox Superfine)	40:60	Spraying	One coat, dried in air. 25 min., entered oven at 175 F., then raised to 350 F. and held ½ hr., cooled to 225 F.	0.4	Good	304 000
No. 19......	Ferhert Scharnoorfer Primer	...	...	...	Spraying	One coat, baked for 25 min. at 325 F.	1.3	Good	515 000
No. 20......	Unichrome Lacquer	...	...	...	Spraying	One coat, baked for 1 hr. at 375 F.	1.0	Slight blistering	582 000
No. 21......	Corrosite	...	...	...	Spraying	Dried in air	0.9	Fair	707 000
No. 22......	Phenolic and vinyl mixture	80% phenolic resin (Bakelite XV-16607) 20% vinyl resin (Vinylite XYHL)	40% zinc chromate (zinc yellow) 40% iron oxide (Canox Superfine) 20% zinc oxide	40:60	Spraying	One coat, air-dried for 25 min., entered oven at 175 F., then raised to 350 F. for ½ hr., cooled to 225 F. slowly	0.3	Good	828 700
No. 23......	Urea-alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	40% zinc chromate (zinc yellow) 40% iron oxide (Canox Superfine) 20% zinc oxide (ZZZ-22)	40:60	Spraying	One coat, baked for 30 min. at 300 F.	1.6	Very slight pitting	301 700
No. 24......	Urea-alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	100% iron oxide (Canox Superfine)	40:60	Spraying	One coat, baked for 30 min. at 300 F.	1.1	Good	580 600
No. 25......	Urea-alkyd (non-oxidizing)	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	100% carbon (Black Knight Velvet lamp-black)	40:60	Spraying	One coat, baked for 30 min. at 300 F.	1.7	Good	476 500
No. 26......	Two primer coats	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	40% zinc chromate 40% iron oxide 20% zinc oxide	40:60	Spraying	Four coats, 15 min. drying or baking after each coat. System baked 45 min. at 300 F. after last coat	...	...	647 000(26*a*)[a]
	Two finish coats	60% alkyd-type resin solids (Rezyl 99-5) 40% urea formaldehyde-type resin solids (Beckamine P-138)	Nonpigmented	...	Spraying				3 593 500(26*b*)[a]
No. 27......	Two primer coats	Ucilon Primer 405-P	Nonpigmented	...	Spraying	Five coats, 5 min. drying between primer coats and 30 min. before finish coats applied. 30 min. drying between finish coats. System dried 6 days in air before test.	...	...	1 878 400
	Three finish coats	Ucilon White	Nonpigmented	...					

[a] (26*a*) = Specimen No. 26*a* operated 21,895,000 cycles (was Specimen 15) before coating was scratched severely, and 647,000 cycles after scratching.
(26*b*) = Specimen No. 26*b* operated 64,136,800 cycles before coating was scratched severely and 3,593,500 cycles after scratching.

Specimen No. 15*b*, with an identical coating, ran about 120 times normal lifetime in the standard corrodent with no evidence of damage. It was then transferred to a 7 per cent salt solution containing 2 per cent NaOH to determine its ability to resist alkaline solutions. It ran an additional 30 times normal lifetime in the caustic without a coating failure. It was finally scratched severely and operated for an additional 8 times normal lifetime in the standard corrodent before failure. Its total life was, therefore, about 158 times normal lifetime, or a total of 67,730,300 cycles. This represents more than 650 hr. of operation at 1750 rpm. Had the specimen not been purposedly scratched, its lifetime would probably have been very much greater.

Several additional complete coating systems were prepared, including typical commercially available coatings. These specimens have not been tested as yet because there is little hope of causing the coatings to fail in periods less than several months under the standard test conditions. There is no doubt that there are available many different types of organic coating systems which would greatly increase the operating life of test specimens in brine. It also seems apparent that such coating systems, while intact, offer complete protection against corrosion and resultant corrosion-fatigue failures when the applied stresses are below the endurance limit of the steel. The question then becomes one of the relative lifetimes of the coating materials under operating conditions and the performance of the steel after the coating is first broken. For this reason, tests were next undertaken on specimens in which the coatings were purposely scratched through to the underlying steel before the corrosion-fatigue tests were started.

COMPARATIVE TESTS OF ORGANIC COATINGS WITH SCRATCHES PURPOSELY CUT THROUGH THE COATINGS

Several of the coatings previously tested intact were also scratched through to the base metal and given fatigue tests in the standard corrodent (Table IV and Fig. 8). In preparing new specimens for scratch tests, it was found that a more uniform cut through the coating could be obtained by wrapping a single turn of fine piano wire about the specimen at its minimum diameter prior to coating and then removing the wire after the coating had dried.

None of the specimens with scratched coatings had very long fatigue lifetimes. Negligible improvement over the performance of bare specimens was offered by the scratched, single-layer organic coatings. Even the four-coat systems (which performed so excellently when intact) failed, in one case, at only $1\frac{1}{2}$ times normal lifetime when scratched. In general, however, the specimens with thicker coating systems seemed to survive longer. The pigments did not appear to influence the results greatly.

These results indicated that organic coatings offered excellent protection until damaged or penetrated by scratches, but thereafter the operating lifetime was about the same as that of new, bare steel. It was possible that some strengthening by prolonged understressing (fatigue loading below 48,000 psi.) may have occurred while the coatings were intact, resulting in improved performance of the steel. This may account for the relatively long lifetime with a fractured coating observed for Specimen 26*b*.

Since the steel base appeared to be in good condition until the organic coating had been fractured, it is possible that such coatings might be stripped from the steel when the first coating failures occur. The steel might then be recoated and would probably survive for the life of the second organic coating. The recoating process might be continued until the steel was in danger of failure from causes other than corrosion-fatigue of the coated surfaces.

Discussion

Small-scale laboratory corrosion-fatigue tests provide a rapid and economical means of comparing materials and test conditions and permit relatively precise control of test variables. However, due caution is in order before attempting to apply quantitative data from small-scale tests to full-scale equipment. Because of the "size effect," the larger specimens or full-scale assemblies usually tend to fail at shorter lifetimes or at lower stresses than those indicated from small-scale tests. Since corrosion progresses continuously in steels exposed to industrial brines, the long-time effects usually cause failure to occur in industrial equipment after many fewer applications of load than in high-speed laboratory corrosion-fatigue tests. Fortunately, the trends revealed in the laboratory tests, provided these tests duplicate or simulate actual full-scale operating conditions and materials with validity, are usually borne out by full-scale operating experience.

The data presented in the preceding sections of this paper have been subjected to full-scale tests in the oil-well drilling industry. A full-scale, rotating-beam, corrosion-fatigue test of a bare joint of $3\frac{1}{2}$-in. pipe of similar steel was tested in a 3 per cent solution of the same corrodent. It failed at a lifetime identical with that observed for the small test specimen (Fig. 3). Drill strings have been made up with alternate joints of uncoated and commercially coated new pipe, and operated until corrosion-fatigue damage forced the retirement of the individual joints. The organic-coated pipe remained in service at least 3 times as long as the uncoated pipe. Where coating failures have been observed, the steel beneath the coating fracture was found to be clean and in good condition.

Further full-scale tests in other industries are needed to verify or modify the results indicated by the laboratory tests. As practical operating experience is accumulated for steels protected with organic coatings in order to improve corrosion-fatigue operating life, the value of the procedure may be determined and its usefulness extended.

To obtain good adherence of organic coatings on steel surfaces, the steel must be cleaned thoroughly before application. Methods such as sandblasting or shot-peening are finding acceptance in many applications. To avoid premature failures, full coverage must be obtained, with a minimum of pits and blisters. Careful dipping or spraying, or filling and withdrawal of the coating fluids at proper rates may be necessary in order to obtain uniform, nonporous coats. Multicoat systems are preferred, with adequate drying or baking of each coat before the application of the succeeding coat. The final coating should be carefully inspected to insure that it does not contain flaws which might permit corrosive attack to begin locally immediately after the steel is put into use in a corrosive environment. Periodic inspection thereafter may be appropriate to guide removal of units on which coating failures have begun to develop. Throughout the service life of the steel, effort should be made to keep the coating in perfect (impervious) condition. Since no good method of patching a coating is available where the surface of the steel has become contaminated with corrosion products, it will usually be necessary to remove damaged coatings from the steel and replace them with new coatings applied to carefully cleaned surfaces.

In some cases, it may appear desirable to apply organic coatings to steel which has already been subjected to corrosion-fatigue service without protective coatings. Such used steel should be inspected carefully before the coating is applied, and rejected if it has been severely pitted or if fatigue cracks have begun to propagate. The stress concentration effects of such notches, pits, or cracks may permit pure fatigue failures to develop, even though the organic coating prevents any further corrosion.

The use of organic coatings on steel surfaces is not necessarily a guarantee that premature failures will not occur, nor is this method a cure-all. In addition to maintaining the coating in good condition, it is highly important to control the operating stresses. If the operating stress exceeds the endurance limit of the steel, pure fatigue failures may occur even in the complete absence of corrosive attack. Even below the endurance limit, there are obvious advantages in maintaining the operating stress at the lowest feasible levels. The corrosion-fatigue life of uncoated steels is greatly increased if the stress can be reduced. Thus the life after coating failure will be much longer if the stresses are low.

In many cases, it is impossible to coat all of the surfaces which may be subject to corrosive attack. Threaded joints, sliding parts, areas subject to abrasion—these and other localities may not be suitable for the application of coatings. Therefore, it is often advisable to take additional measures, other than coating of accessible exposed areas, to prevent or reduce corrosive attack on the steel. Such measures might include the addition of chemical inhibitors to the brine, the removal of corrosive components from the environment, avoidance of dangerously corrosive concentrations where possible, sealing of uncoated areas (as in threaded joints) from the corrosive fluid, periodic cleaning, and other procedures appropriate to the situation.

Although protective organic coatings can be designed with remarkable stability and durability, they can be severely damaged by abuse. Few coatings can survive sharp blows or cuts from hammers, other steel objects, and so on. Neither can they survive heat such as is produced during welding. For this reason, extreme care to avoid fracturing organic coatings during fabrication, transportation, assembly, maintenance, and inspection is essential if the coating is to serve its purpose of prolonging the useful life of steel under corrosion-fatigue service conditions. The coating is no stronger than its weakest point; a single crack or fracture may be the focal point where corrosive attack can produce a nucleus for fatigue failure.

CONCLUSIONS

It appears possible that organic coatings may provide a great help in preventing premature corrosion-fatigue failure of steels in industrial equipment, even when the steels are subjected to severely corrosive brines or alkali solutions. The protection offered by the organic coatings is effective so long as the properly designed coating system is intact and impervious to the corrodent. When the coating fractures, and the corrodent is permitted to contact the steel, the effective protection ceases and corrosion begins. Thereafter, the steel may be expected to operate little or no longer than now, uncoated steel would survive under identical conditions. With extremely corrosive brines and solutions which supply copious quantities of new corrodent to the steel surface, the inhibitive pigments in the primer coats cannot be expected to provide long protection against corrosion when the coating is scratched or fractured.

REFERENCES

(1) L. R. Jackson, H. M. Banta, and R. C. McMaster, "Investigation of Causes of Drill-Pipe Failures in the Permian Basin," *The Drilling Contractor*, Vol. 2, No. 3, May 15, 1946, pp. 43–51.

(2) F. N. Speller, I. B. McCorkle, and P. F. Mumma, "Influence of Corrosion Accelerators and Inhibitors on Fatigue of Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 159 (1928).

(3) L. R. Jackson, H. M. Banta, and R. C. McMaster, "Laboratory Corrosion-Fatigue Tests of Steels," *The Drilling Contractor*, Vol. 3, No. 1, November 15, 1946, pp. 46–60.

(4) L. R. Jackson, H. M. Banta, and R. C. McMaster, "Battelle Reports on Field Survey," *The Drilling Contractor*, Vol. 3, No. 3, April 15, 1947, pp. 42, 47, 55.

(5) L. R. Jackson, H. M. Banta, and R. C. McMaster, "Periodic Cleaning of Drill Pipe," *The Drilling Contractor*, Vol. 3, No. 4, June 15, 1947, pp. 50–53.

(6) L. R. Jackson, H. M. Banta, and R. C. McMaster, "Use of Chromate Additions in Drilling Fluids," *The Drilling Contractor*, Vol. 3, No. 5, August 15, 1947, pp. 77–82.

(7) L. R. Jackson, H. M. Banta, R. C. McMaster, "Possibilities of Zinc Coatings on Drill Pipe," *The Drilling Contractor*, Vol. 3, No. 6, October 15, 1947, pp. 50–51.

(8) W. H. Swanger and R. D. France, "Effect of Zinc Coatings on the Endurance Properties of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 430 (1932).

(9) D. G. Watt, "Fatigue Tests on Zinc-Coated Steel Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 717 (1940).

(10) J. N. Kenyon, "A Corrosion-Fatigue Test to Determine the Protective Qualities of Metallic Platings," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 705 (1940).

(11) T. J. Dolan and H. H. Benninger, "The Effect of Protective Coatings on the Corrosion-Fatigue Strength of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 658 (1940).

(12) L. A. Jordan and L. Whitby, "The Preservation of Iron and Steel by Means of Paint," Sixteenth Bulletin of the Research Association of British Paint, Colour, and Varnish Manufacturers, Paint and Research Station, Middlesex, England, p. 68.

(13) D. G. Sopwith and H. J. Gough, "Effect of Protective Coatings on the Corrosion Fatigue Resistance of Steel," Vol. 35, pp. 315-319 (1937).

(14) R. D. Barklie and H. J. Davies, "The Effect of Surface Conditions and Electrodeposited Metals on the Resistance of Materials to Repeated Stresses," *Proceedings*, Am. Inst. Mechanical Engrs., pp. 731–750 (1931).

DISCUSSION

Mr. H. R. Copson.[1]—Can you stop the corrosion by keeping air out of the brine?

Mr. R. C. McMaster (*author*).—I don't believe so. We did not make any effort to remove the air from the brine. The brine used in the field has lots of opportunity to pick up air. It is stored in tanks on the surface of the ground and sent through pumps and filters on the way down. There is probably plenty of oxygen available. Secondly, in our tests we did try the test with totally immersed specimens. Their lifetimes were slightly shorter than those of specimens tested with the corrodent dropping upon them in the presence of excess air. The two points shown as squares on the black *S-N* curve characterized the totally immersed specimens.

I do believe, of course, that it would be desirable to remove oxygen if possible since it is a fundamental factor in corrosion, it would be fine. We did not have any way of removing it, in the field.

Mr. R. C. Bowden, Jr.[2] (*by letter*).—Mr. R. C. McMaster of Battelle Memorial Institute has indicated an impressive increase in the resistance of S.A.E. 1045 steel to corrosion-fatigue to be attributed to a nonmetallic coating. At 40,000 psi. an increase in life from 430,000 cycles or 4 hr. to 64,000,000 cycles or 600 hr. is noteworthy, although 4 hr. seems to be a rather brief period for any serious corrosion to occur. The Research Laboratory of National Tube Co. has done some work on protective coatings in relation to corrosion-fatigue which parallels the work done by Mr. McMaster. This work, some of which has been published,[3] shows that the endurance life of steel in corrosive brines can be lengthened by protective coatings if the coatings are kept intact. When the coating is broken, however, we have found that oxygen concentration cells form at bare spots making the stressed metal at these points more anodic, tending to break down the adjacent protective film and causing localized corrosion. When this takes place, the corrosion endurance life of the test specimen may be less than that of an uncoated test under the same testing conditions.

The question also arises as to other tests on the coating that may have an effect on the length of life of the coating. L. E. Trishman[4] lists a set of requirements for coatings for drill pipe. In the tests run at the Research Laboratory we first immerse the specimens, after they have been coated and dried, in the corrosive brine for a period of time sufficient to saturate the coating (as much as possible) before actual fatigue testing is started. This varies from 6 to 40 days. By this method we try to evaluate the coating as to its ability to stand up under corrosive conditions as well as its resistance to corrosion fatigue.

---

[1] Research Chemist, The International Nickel Co., Inc., Research Laboratory, Bayonne, N. J.

[2] Research Laboratory, National Tube Co., Pittsburgh, Pa.

[3] F. N. Speller and McCorkle, *Corrosion Fatigue and Protective Coatings*, Fourteenth Annual Meeting, Am. Petroleum Inst., Chicago, Ill., October 24, 1933.

[4] L. E. Trishman, *Plastic Coating Inside Surface of Drill Pipe to Combat Corrosion Fatigue Failures*, The Petroleum Engineer, November 1946, p. 194.

We have not been able to find, up to this time, any plastic coating which is perfect, that is, impervious to moisture, and which will not crack or scratch.

I should like to compare the methods used by Mr. McMaster in this report to the methods used at the National Tube Co. Research Laboratory. We feel that the speed used by Mr. McMaster (1750 rpm.) is too fast. We have found that speed has a noticeable influence on fatigue properties under reversal of stress testing when corrosion is present, and for this reason have consistently adhered to the speed of 189 rpm. in corrosion-fatigue even though this requires 40 days to run the conventional 10,000,000 cycle test. The constant speed of 189 rpm. was chosen to more nearly simulate the stress-time cycle encountered in field practice. When a sample is run at 1750 rpm. at a high stress, it may break in a few hours or days which hardly seems enough time for sufficient corrosion to take place even under the influence of cyclic stress.

We feel that the stress range used (40,000 psi. for the steel involved) is too high for corrosion-fatigue. We have found that the results of specimens in corrosion-fatigue when under a load close to the air endurance limit are erratic and in some cases so high as to be misleading. According to our findings, a stress range close to the corrosion endurance limit of 10,000,000 cycles (at 189 rpm.) must be used if the tests are to have any practical value. Mr. McMaster states that the corrodent had little effect upon the operating life of uncoated steel, at stresses above 48,000 psi. when tested at 1750 cycles per min. This is probably due to the combined effect of the previously-mentioned factors; namely, insufficient time for corrosion to take place and erratic effect caused by high stress.

The brine used at the National Tube Co. Research Laboratory is more corrosive (on the basis of secondary salinity) than the 7 per cent brine used at Battelle Memorial Institute. We use a 30,000 ppm. brine (22,000 ppm. NaCl, 5000 ppm. $CaCl_2$, and 3000 ppm. $MgCl_2$) which was chosen as an average composition of several corrosive oil well waters. The brine is circulated over the specimen in a system open to the atmosphere since we feel that the mud circulated in a well contains oxygen which has been absorbed in settling tanks and during circulation in the well. As a matter of information, when these factors and conditions are considered, we arrive at a corrosion endurance limit (10,000,000 cycles) of 10,000 psi. for Grade D steel (C – 55) in the uncoated state.

In conclusion, we feel that the problem of corrosion-fatigue is quite complicated and does not have a simple answer. Coatings offer some protection, but under certain conditions they may crack or become scratched, thus subjecting unprotected steel to corrosive attack. Many experiments have been made with inhibitors, but the results have been quite erratic, and due to the large quantities required in practice the use of inhibitors is not economical. Changes in steel-making practices or the development of compositions which will have higher resistance to corrosion should assist in providing the answer.

As long as corrosion-fatigue is a pertinent factor in drilling operations, it is necessary to study means of combating the problem. Research projects are under way investigating the above as well as other subjects, such as the effect of cathodic protection with outside electrical currents and by the use of sacrificial anodes.

MR. ROBERT C. MCMASTER (*author's closure by letter*).—Mr. Bowden's highly informative discussion lists an excellent series of corrosion-fatigue test conditions which should provide very useful data in long-time tests. We chose a moderately high stress (80 per cent of the endurance limit in air) and a moderately high speed (1750 rpm.) for the tests at Battelle in order to obtain the desired information economically in the limited time available for the research. Quantitative lifetime data are seldom of significance in corrosion-fatigue testing, save for the trends they reveal. The trends shown, and the conclusions based thereupon, were the same for the Battelle tests as those given by Mr. Bowden for the much longer tests at the National Tube Co. We both agree that intact impervious organic coatings protect the steel from corrosion (and consequent corrosion-fatigue failures) but that when the coating fractures, and the corrodent is permitted to contact the steel, deterioration may be rapid, and the failure may occur as rapidly as with new, uncoated steel.

We are particularly interested in Mr. Bowden's comment that changes in steelmaking practice or the development of compositions which will have higher resistance to corrosion should assist in providing the answer to the problem of corrosion-fatigue. Such a development in drill pipe steels would be welcomed by operators drilling the hard rock formations under the corrosive conditions found in the Permian Basin oil fields. Until such improved steels are made available, however, the operator may obtain some improvement in performance of available grades of drill pipe by a proper use of organic coatings.

AN EXPERIMENTAL STUDY OF THE INFLUENCE OF VARIOUS FACTORS ON THE MODE OF FRACTURE OF METALS*

By P. G. Jones[1] and W. J. Worley[1]

Synopsis

The factors related to the external conditions imposed on a given metal which determine whether the mode of fracture will be a ductile type or a brittle type are considered to be rate of strain, stress concentration, state of stress, and temperature. This paper presents experimental results on the influence of combinations of these factors on the mode of fracture of three steels and an aluminum alloy. The effect of strain aging on the mode of fracture of a semi-killed steel and a rimmed steel was also investigated.

In the tests, the stress concentration and state of stress were varied by using notched and unnotched specimens in tension, and unnotched specimens in torsion. The rate of strain was varied by making static and impact tests in tension and in torsion. Temperatures from room temperature to −310 F. were used in determining the temperature of transition from a ductile to a brittle type of fracture.

One mode of fracture of a ductile metal member may be designated as a ductile type of fracture by which is meant that considerable plastic deformation precedes the fracture. This plastic deformation or yielding may involve a considerable portion of the member and a relatively large amount of energy is absorbed before fracture occurs. The plastic deformation which precedes the fracture usually is accompanied by strain hardening of the metal (also designated by work hardening or cold working) so that the fracture that finally results is not the fracture of the original metal but rather of a metal whose properties are in the process of changing until fracture occurs.

Another mode of fracture of a so-called ductile metal member may be designated as a brittle type of fracture by which is meant that neglibible plastic deformation preceded the fracture; such a fracture usually has a granular or crystalline appearance and the energy absorbed may be relatively small. A brittle type of fracture is likely to occur if the metal is subjected simultaneously to (1) a high rate of straining, (2) a non-uniform stress distribution (stress concentration), (3) a triaxial state of stress (principal stresses in three directions), and (4) a low temperature. The embrittling effect of any one of these factors may not be independent of the effect of other factors. High stress concentrations, for example, are probably accompanied by a triaxial state of stress and a high localized rate of strain.

The influence of certain combinations of the foregoing factors on the two modes of fracture has been studied by a number of investigators, and several theories have been proposed to explain the mechanism of brittle fracture. However, no theory of fracture has proved to be a satisfactory explanation of brittle behavior. The purpose of this paper is not to present a theory or explanation of brittle fracture, but rather to reexamine the effect of combinations of some of the conditions imposed by previous investigators and to extend our knowledge of the embrittling effect of a wider range of combinations of conditions.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Assistant Professor and Instructor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

TABLE I.—CHEMICAL COMPOSITIONS OF METALS BY PER CENT.

	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Zinc	Copper	Magnesium	Chromium	Iron	**Aluminum**
Semi-killed steel	**0.13**	**0.45**	0.012	**0.030**	**0.16**	...	...	...	...	**rem.**	...
Rimmed steel	0.17	0.35	0.016	0.034	0.008	...	...	...	...	rem.	...
S.A.E. 1095 steel[a]	0.97	0.40	0.04	0.05	...	...	...	...	...	rem.	...
75S-T aluminum alloy	...	0.15	...	...	0.25	**5.6**	1.6	2.5	0.26	0.35	**rem.**

[a] Nominal composition.

The factors previously mentioned relate to the external conditions imposed on a given metal or a metal in a given state or condition. Other factors that play a part in the embrittling action of a metal are associated with its internal state which is closely connected with its life history; they include inherent or residual stresses, internal discontinuities and cracks, grain size, grain orientation, strain hardening or cold working, state of deoxidation, strain aging, etc. These factors were introduced in the experimental work herein reported only to a minor degree by subjecting a semi-killed steel and a rimmed steel to strain aging and then imposing combinations of the previously mentioned external factors on the two steels.

It has been proposed to make a distinction between ductile and brittle types of fracture by designating a ductile fracture as a shear fracture and a brittle fracture as a cleavage fracture. This nomenclature has been used to a considerable extent in describing fractures of wide specimens of ship plate, and the type of fracture is determined by visual observation of the fractured surfaces of the plate. If the fractured surface shows evidence of shear deformation, in which a sliding action of the metal on slip planes occurs, the fracture is termed a shear type of fracture. If the fractured surface shows evidence that the plane of separation is the cleavage plane for the crystal, such as the (100) planes in ferrite crystals, the fracture is termed a cleavage type of fracture. All cleavage fractures, however, are not brittle fractures, since it is possible to obtain a cleavage fracture after considerable plastic deformation has occurred. Furthermore, all brittle fractures are not cleavage fractures since separation may occur in the grain boundaries. It would appear, therefore, undesirable to use these terms to distinguish between ductile and brittle types of fracture.

It would seem that a more useful and satisfactory explanation of the transition from ductile to brittle behavior of metal will be found by considering the metal to be composed of two component parts (two phases), one part or phase consisting of the ordered crystalline phase and the other the unordered phase composed of the grain boundary material. The response of these two phases to temperature, rate of strain, etc., are known to be quite different at different temperatures, strain rates, etc. But until research in this field is carried further, the prediction of the influence of various combinations of the embrittling factors will be unsatisfactory, and reliance must be placed on the results of more extensive tests. Furthermore, until a more satisfactory explanation of the phenomenon of fracture is available, proposed attempts to correlate transition temperatures at which brittle fractures occur at lower temperatures under single-blow impact loads with fracture under repeated loads or in long-time load-to-fracture tests at elevated temperatures will probably prove to be of doubtful basic value.

TABLE II.—OUTLINE OF TESTS TO DETERMINE THE INFLUENCE OF VARIOUS FACTORS ON THE MODE OF FRACTURE OF A METAL.

Rate of Strain in Test	Degree of Triaxiality; Indicated by Ratio of Maximum Tensile Stress to Maximum Shearing Stress, σ/τ			Temperature
Very slow (ordinary testing machines with load gradually applied)	Torsion Test		$\sigma/\tau = 1$ (very small). Unnotched specimen	−310 F. to room temperature
	Tension Test	(a)	$\sigma/\tau = 2$ (small). Unnotched specimen	same
		(b)	σ/τ = medium. Shallow notched specimen with root of notch not too sharp. σ = large	same
		(*c*)	σ/τ = large. Deep notch. τ = very small	same
Medium (Charpy tension impact and torsion impact)	Same as torsion test above			same
	Same as tension test (*a*) above			same
	Same as tension test (*b*) above			same
	Same as tension test (*c*) above			same
Relatively high (high velocity pneumatic type tension impact machine)	Same as tension test (*a*) above			same
	Same as tension test (*b*) above			same
	Same as tension test (*c*) above			same

MATERIALS

The materials used for the tests included a semi-killed steel, a rimmed steel, S.A.E. 1095 steel, and an aluminum alloy 75S-T. The compositions of these metals are given in Table I. The three steels were received in the form of round hot-rolled bars $\frac{3}{4}$ in. in diameter. The 75S-T aluminum alloy was $\frac{7}{8}$ in. in diameter. The S.A.E. 1095 steel was annealed for 1 hr. at 1400 F. and cooled in the furnace. The other metals were tested in the as-received condition.

To determine the effect of strain aging, bars of the semi-killed steel and the rimmed steel were stretched 4 per cent in a tension testing machine. Specimens were then machined from these bars, taking care not to overheat the material and were tested within 4 hr. after prestraining in order to find the effect of straining without aging. Other bars were stretched 4 per cent and placed in a 500 F. oven for 45 min. to accelerate the aging process. Specimens were then machined from these bars and tested.

TEST METHODS

An outline of tests to determine the influence of various factors on the mode of fracture of a metal is given in Table II. This outline of tests was suggested in a paper by F. B. Seely (1)[2]. The purpose of this table is to give a brief summary of the factors which relate to the external conditions imposed on a given metal or a metal in a given state or condition and to indicate the method of varying these factors. A more detailed discussion of these factors will be given in the subsequent paragraphs.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 662.

The effect of stress concentration and of state of stress on the mode of fracture were obtained by testing cylindrical tension specimens containing circumferential V-notches. Figure 1 shows qualitatively the distribution of stress (as given by Neuber (2) for a notch of hyperbolic profile) in a rather sharply notched tension specimen under elastic conditions. At the root of the notch a relatively high concentration of longitudinal stress (σ_1) occurs and the radial stress (σ_2) is zero. Hence the maximum shearing stress at this point on 45-deg. planes is also large ($\tau_{\text{max.}} = \frac{1}{2}\,\sigma_1$). At the center of the notch the radial stress (σ_2) is equal to the transverse stress (σ_3), and the longitudinal stress (σ_1) is much less than its value at the root of the notch. The maximum shearing stress ($\tau_{\text{max.}} = \frac{1}{2}\,(\sigma_1 - \sigma_2)$) is thus small at the center and at all other points except those near the root of the notch, and hence embrittlement is promoted by the notch since yielding is associated with high shearing stresses.

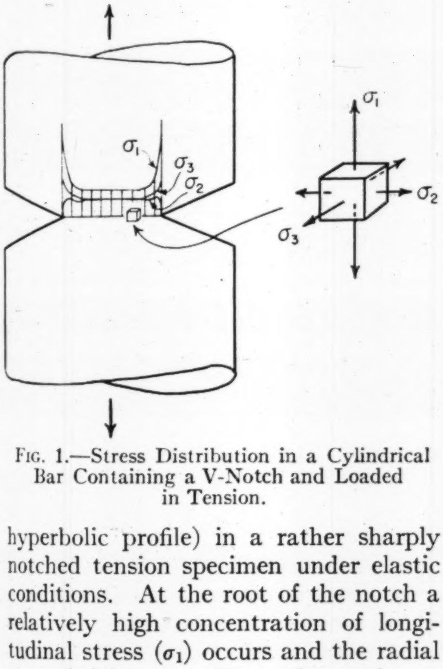


FIG. 1.—Stress Distribution in a Cylindrical Bar Containing a V-Notch and Loaded in Tension.

A specimen having a relatively deep notch and a small notch angle contains relatively high radial and transverse stresses compared to the longitudinal stress; thus the degree of triaxiality is large and the maximum shearing stress is small. A similar specimen containing a shallow notch will have a higher stress concentration at the root of the notch but the degree of triaxiality for the shallow notch is less than that of the deep notch. Thus one way to obtain a relatively high degree of triaxiality is to use a deep-notched specimen; and one way to obtain a high stress concentration with a low degree of triaxiality is to use a shallow-notched specimen.

A uniform stress distribution and a uniaxial state of stress may be obtained by using unnotched tension specimens. Since the maximum shearing stress for a uniaxial state of stress is one half the longitudinal stress ($\tau_{\text{max.}} = \frac{1}{2}\,\sigma_1$), this state of stress is more favorable for plastic deformation than is a triaxial state of stress. A biaxial state of stress, in which one of the principal stresses is a compressive stress, may be obtained by using an unnotched solid torsion specimen. Since the maximum shearing stress in torsion equals the maximum principal stress ($\tau_{\text{max.}} = \sigma_1$), this state of stress is the most favorable for plastic deformation.

All of the notched tension specimens used in the investigation reported in this paper had a diameter of 0.2 in. at the root of the notch. The radius of curvature at the root of the notch was 0.006 in. and the notch depth varied from 0.015 in. to 0.150 in. The notch angles used varied from 45 to 150 deg. The notches were turned in a lathe with a specially ground tool whose contour was frequently checked in an optical projection machine. After machining, the specimens were polished at the root of the notch with thread and fine grinding compound. Measurements of the notch contour and of the root diameter were then found with the optical projection machine. The unnotched tension specimens tested had a diameter of 0.2 in., and the parallel section was 1 in. long except for the Charpy specimens for which the parallel section was 2 in. long. The specimens were finished slightly undersize in the center to insure the specimens breaking near the center. The torsion specimens were 0.25 in. in diameter and the parallel section was 1.25 in. long.

The effect of rate of strain on the mode of fracture in tension was obtained by making tests in a static tension testing machine, a Charpy impact tension machine, and a pneumatic type high-velocity impact tension machine. By a static testing machine is meant a machine which strains the specimen at a relatively low rate. The nominal speed of head in the static tension tests was 0.05 in. per min. and the tests were made on specimens having threaded ends.[3] The Charpy impact machine used has a capacity of 223 ft-lb. and a striking velocity of 17.4 ft. per sec. A special tension attachment on the Charpy machine was used for the tension-impact specimens by means of which a close approach to axial loads was

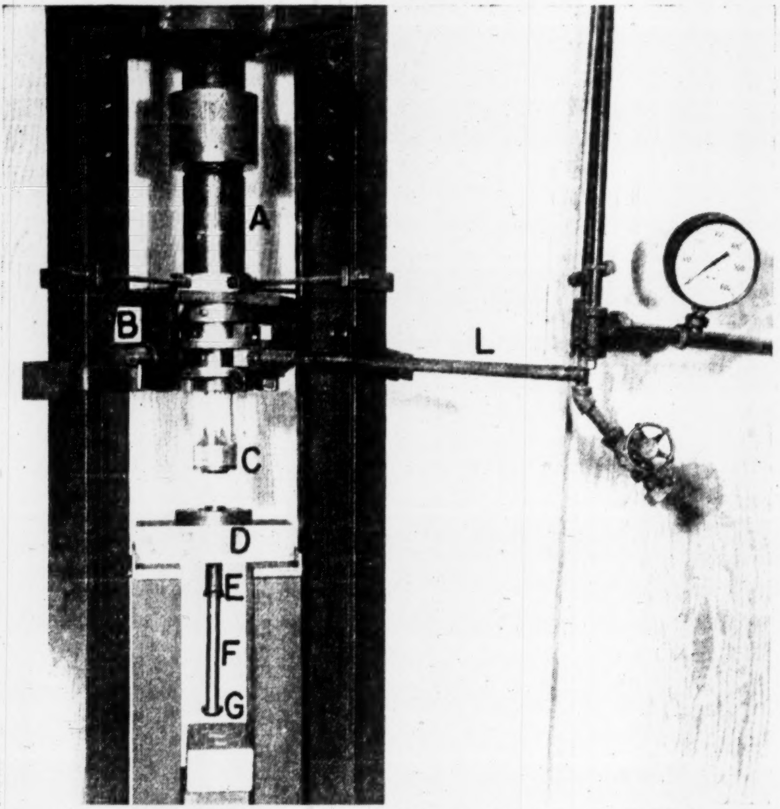


FIG. 2.—Pneumatic Type, High-Velocity Tension Impact Machine.

---

[3] The actual rate of strain in the specimen is influenced to a considerable extent by the method of gripping the specimen (**3**).

produced. The specimens had shouldered ends.

The pneumatic type high-velocity impact tension machine is shown in Fig. 2. This machine was originally designed by N. M. Newmark in an investigation sponsored by the National Defense Research Committee, and was used for bending impact tests on reinforced-concrete beams. For the investigation reported in this paper, the machine was adapted to impact tension tests at low temperatures. The tension specimens used had threaded ends, and one end of the specimen is screwed inside the hollow bar *F* and the other end is attached to the tup *G*. For low temperatures, the bar and specimen are immersed in a refrigerant which is removed immediately before the specimen is tested. The hammer *C* is held in the high-pressure cylinder *A* by means of the pins *B* and the air pressure in the cylinder is increased to the desired value. The pins are then withdrawn by means of a cam attached to the lever *L* and the hammer shoots down and hits the plunger *E* which slides down bar *F* hitting the tup *G* and thus breaking the specimen. The kinetic energy of the hammer *C* is absorbed by a lead ring placed on the block *D*. The velocity of the plunger at the instant it strikes the tup was found to be 175 ft. per sec. for

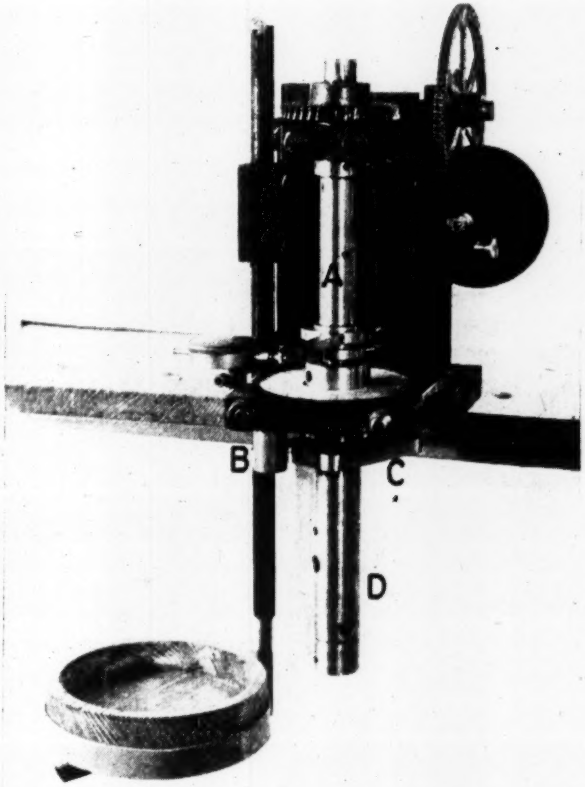


FIG. 3.—Static Torsion Machine.

the cylinder pressures used. The kinetic energy of the broken specimen and tup after fracture is absorbed by a lead block placed under the tup.

The effect of rate of strain on the mode of fracture in torsion was obtained by making tests in a static torsion machine and in a torsion impact machine. The static torsion machine is shown in Fig. 3. The specimen is placed inside the tube *D*, one end being attached to the bottom of the tube and the other end to an extension of the lower end of the dynamometer *A*. The top of the tube *D* is attached to the crossbar *C* which is prevented from turning by coming into contact with the pins *B*. For low-temperature tests the tube *D* and the specimen are immersed in a refrigerant and cooled to the proper temperature. The speeds used changed the angle of twist in the specimen at the rate of 0.5 rpm.

The torsion impact machine is shown in Fig. 4. The specimen is placed inside the hollow cylinder *E*, one end being attached to the right end of the hollow cylinder and the other end to the crossbar *D*. When the flywheel is brought up to a speed of 1200 rpm., the button *B* is pressed and a trigger mechanism releases the pins *C* which engage the crossbar, thus breaking the specimen. The speed of the flywheel before and after breaking the specimen is indicated by the tachometer *A*, and the loss in kinetic energy of the flywheel is computed from these readings. When the flywheel is rotating at 1200 rpm., the kinetic energy is 514 ft-lb. When making tests at low temperatures, the specimen and assembly to which it is attached is rotated about the axis *F* so that the specimen may be immersed in a refrigerant. When the specimen is cooled to the proper temperature, the assembly is quickly rotated to the testing position and the lever *G* pushes it to the left where it is secured by the wedge *H*.

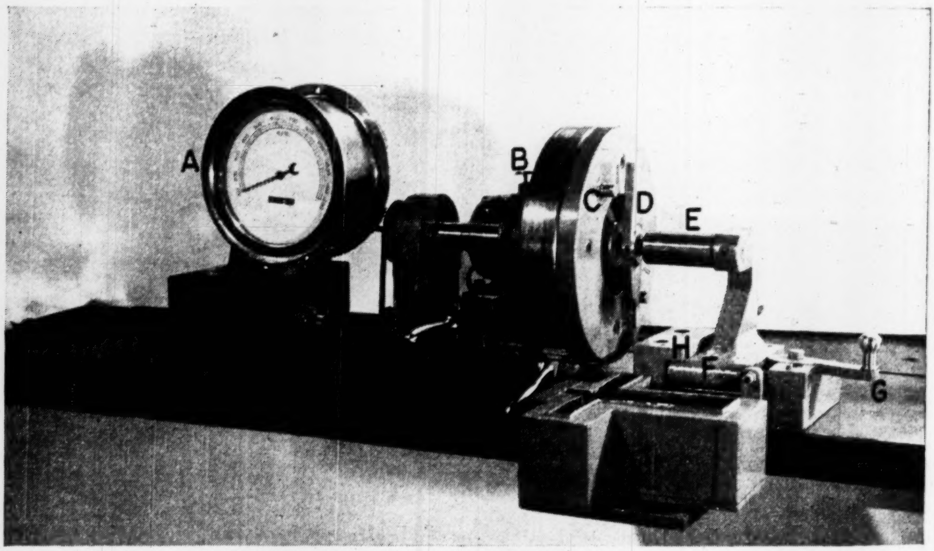


FIG. 4.—Impact Torsion Machine.

Temperatures from room temperature to −100 F. were obtained by using an acetone-dry ice bath. Temperatures from −100 F. to −310 F. were obtained in some tests by using a bath of petroleum ether cooled to the desired temperature by liquid air. However, because of the hazards involved, this method was abandoned and a vapor bath consisting of the vapor from the liquid air was used. The specimen temperature in these tests was measured by a thermocouple attached to the specimen and the temperature was controlled by passing a current through a resistance wire which was wrapped around the specimen but insulated from it. In these tests, it was possible to control the variation in temperature as measured by the thermocouple to within ±2 F. It is probable, however, that the temperature gradient along the length of the specimen and through the cross section of the specimen was greater in these tests than in those in which a liquid bath was used.

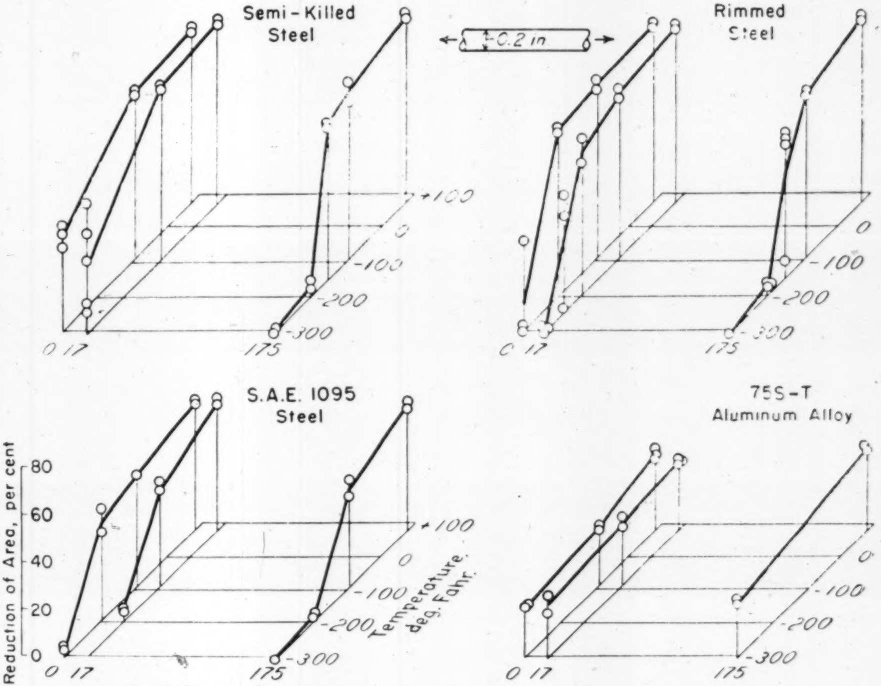


FIG. 5.—Effect of Temperature and Rate of Strain on Reduction of Area of Unnotched Tension Specimens of Four Metals.

RESULTS OF TESTS

In these tests the reduction of area is used as the criterion in determining the mode of fracture of a metal for any combination of external embrittling conditions, the fracture being considered brittle if the reduction of area in per cent of the original area is less than 3.

The mode of fracture could be determined by a consideration of the energy absorbed by the specimen rather than the reduction of area, but if the fracture is of the ductile type, a comparison of energies absorbed by different specimens may not lead to the same conclusions as a comparison of reductions of areas. The energy absorbed depends on both load and deformation, and since the load may fall off as deformation takes place, the total energy absorbed will not necessarily be large for a large reduction of area. The energy absorbed by the specimen was measured in all of the Charpy impact tests and in some of the static tension tests made at room temperature and at low temperatures in which stress-strain diagrams were drawn. These results are not reported in this paper.

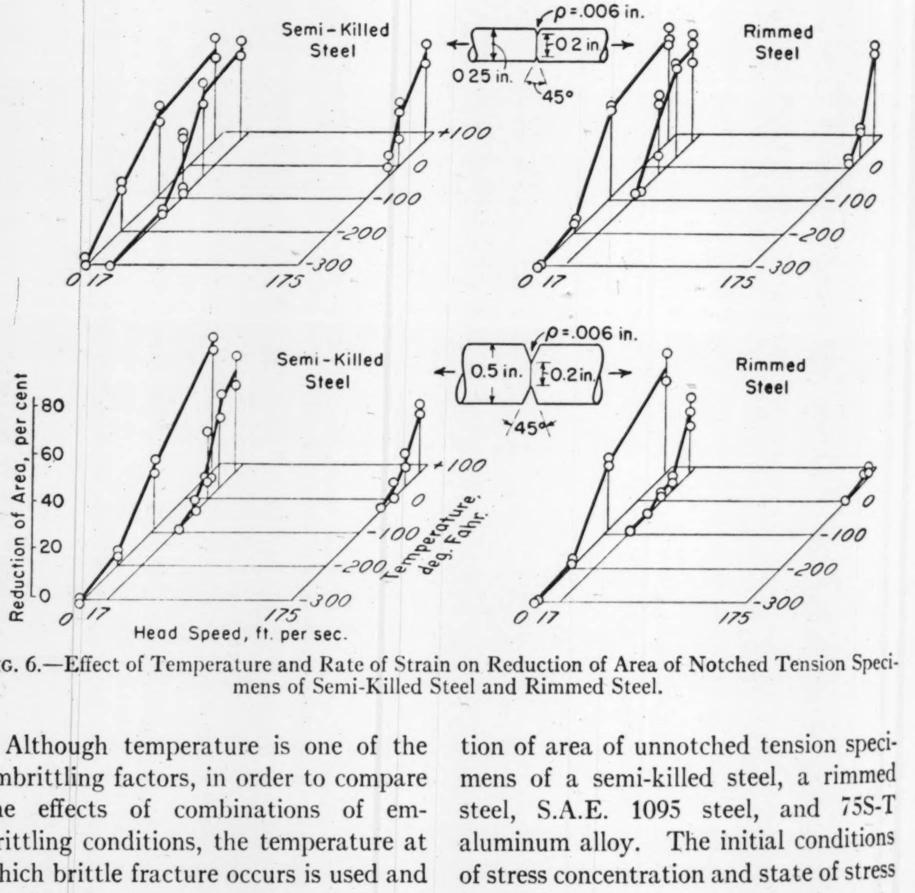


FIG. 6.—Effect of Temperature and Rate of Strain on Reduction of Area of Notched Tension Specimens of Semi-Killed Steel and Rimmed Steel.

Although temperature is one of the embrittling factors, in order to compare the effects of combinations of embrittling conditions, the temperature at which brittle fracture occurs is used and is referred to as the transition temperature. For some combinations of conditions, the transition temperature is not sharply defined since the transition from a ductile fracture to a brittle fracture occurs over a wide range of temperature. An indication of the effect of any combination of embrittling conditions on the transition temperature was obtained by plotting three-dimensional charts in which the reduction of area in per cent of the original area is plotted against the varying temperature along one axis and the varying rate of strain (in terms of head speed) along another axis.

In Fig. 5 is shown the effect of temperature and rate of strain on the reduction of area of unnotched tension specimens of a semi-killed steel, a rimmed steel, S.A.E. 1095 steel, and 75S-T aluminum alloy. The initial conditions of stress concentration and state of stress imposed on the specimens in the tests represented by these data include a uniform stress distribution and a uniaxial state of stress. For these conditions of stress concentration and state of stress, it was possible to find combinations of low temperatures and high rates of strain which would produce brittle fracture in each of the three steels.

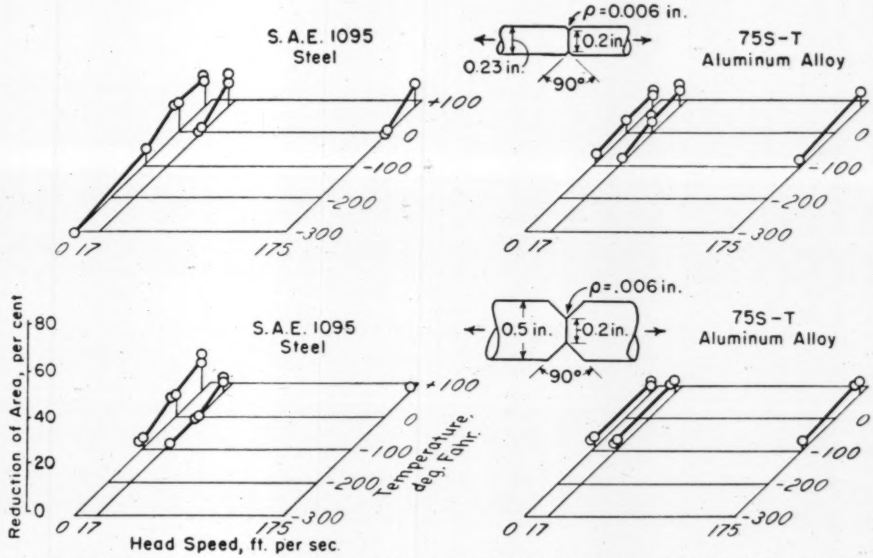


FIG. 7.—Effect of Temperature and Rate of Strain on Reduction of Area of Notched Tension Specimens of S.A.E. 1095 Steel and 75S-T Aluminum Alloy.

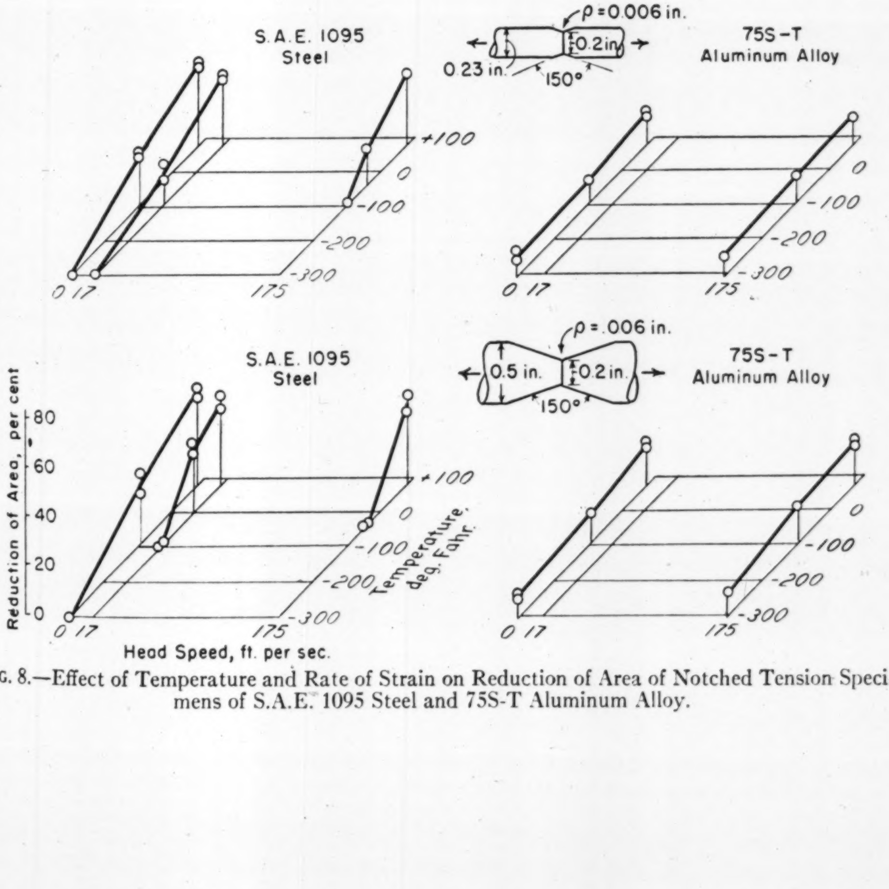


FIG. 8.—Effect of Temperature and Rate of Strain on Reduction of Area of Notched Tension Specimens of S.A.E. 1095 Steel and 75S-T Aluminum Alloy.

However, the low temperatures and high rates of strain used did not produce brittle fracture in the aluminum alloy for conditions of uniform stress distribution and a uniaxial state of stress. For low rates of straining of specimens of the semi-killed and rimmed steels, the temperature of transition from a ductile to a brittle fracture was apparently below −310 F.

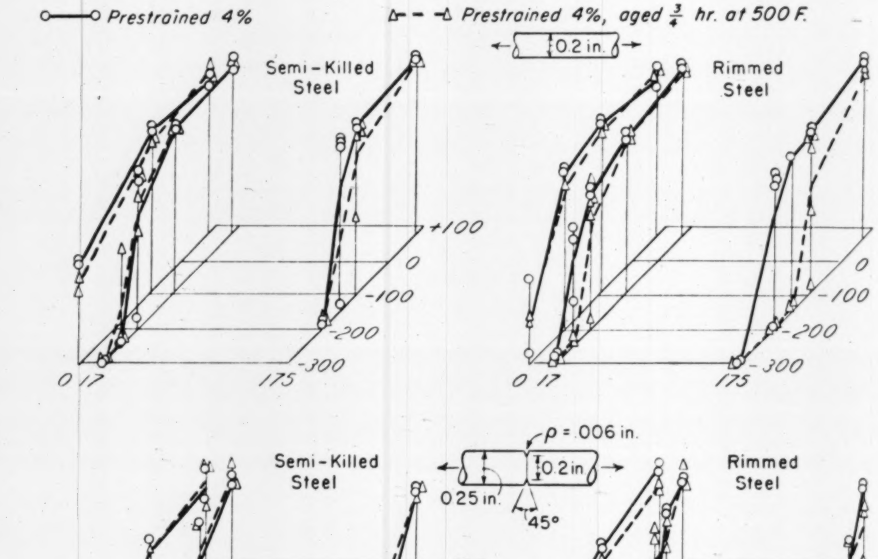


FIG. 9.—Effect of Temperature and Rate of Strain on Reduction of Area of Semi-Killed Steel and Rimmed Steel when Prestrained 4 per cent and when Prestrained 4 per cent Followed by Aging ¾ hr. at 500 F.

There are conflicting opinions among investigators in regard to the effect of rate of strain on the ductility of metals. Some investigators (**4, 5, 6**) have found that unnotched tension specimens of many metals when tested in impact at room temperature and at high temperatures exhibit greater ductility than similar specimens when tested under static loads. Some of the data shown in Fig. 5 are consistent with these findings. However, the conclusion has been drawn (7, 8) that increasing the rate of strain of tension specimens above that of a static test does not promote brittle fracture unless the specimen is notched or the rate of strain approaches bullet velocities. While this may be true at room temperatures, the data shown in Fig. 5 indicate that brittle fracture of unnotched specimens of some steels may occur at rates of strain which are not extremely high if the temperature is sufficiently low; whereas a ductile type of fracture may occur at such low temperatures if the rate of straining is low. This behavior is not typical of metals in general, however, since the non-ferrous metal tested showed ductile fractures at high speeds and low temperatures.

The results of tests of notched tension specimens of the four metals investigated are shown in Figs. 6, 7, and 8. The influence of stress concentration and state of stress on the temperature of transition from a ductile to a brittle type of fracture is noticeable for the steels in that for all combinations of stress concentration, state of stress, and rate of strain the transition temperature is higher than that for corresponding rates of strain in tests of unnotched specimens. The transition temperature for notched specimens of the aluminum alloy is not within the range of temperatures used; however, the deleterious effect of the notch on the ductility of this metal is evident.

The manner in which notch depth, notch angle, and root radius affect stress distribution and state of stress in a cylindrical rod containing a V-notch and subjected to tension is discussed by McAdam and collaborators (9). It is a difficult problem to separate the effects of stress concentration or stress gradient from the effects of state of stress on the mode of fracture if notched specimens are used, since any change in shape of member which changes the stress concentration will also change the state of stress. As shown by Sachs and Lubahn (10), a small plastic deformation (2 per cent or more) will decrease the stress concentration at the root of the notch to an approximately uniform distribution of stress. In all of the ductile fractures and many of the comparatively brittle fractures obtained in this investigation, the ductility exceeded 2 per cent. It would therefore appear that state of stress would have more influence on the mode of fracture than would stress concentration. In Figs. 6, 7, and 8, the transition temperature is higher for the deep-notched specimens than for shallow-notched specimens when tested at corresponding rates of strain. The results in these figures, however, show that for combinations of temperature and rate of strain which produce ductile fractures, the ductility is usually higher for the deep-notched specimens than for shallow-notched specimens. These results are consistent with results reported by other investigators (9), and the explanation is thought to involve the high strain gradient which exists in the shallow-notched specimen.

In Fig. 9 is shown the effect of temperature and rate of strain on the reduction of area of notched and unnotched specimens of a semi-killed steel and a rimmed steel when prestrained 4 per cent and when prestrained 4 per cent followed by aging 45 min. at 500 F. The results indicate that aging has had negligible influence on the transition temperature of the semi-killed steel and has increased the transition temperature of the rimmed steel only to a minor degree for some combinations of stress concentration, state of stress, and rate of strain. It is probable that tests made on the full size $\frac{3}{4}$-in. bars of rimmed steel in the as-rolled condition, when strain aged, would show higher transition temperatures. The rim of relatively pure iron would then be included, and it is thought that the rim is more susceptible to strain aging than is the core of a rimmed steel.

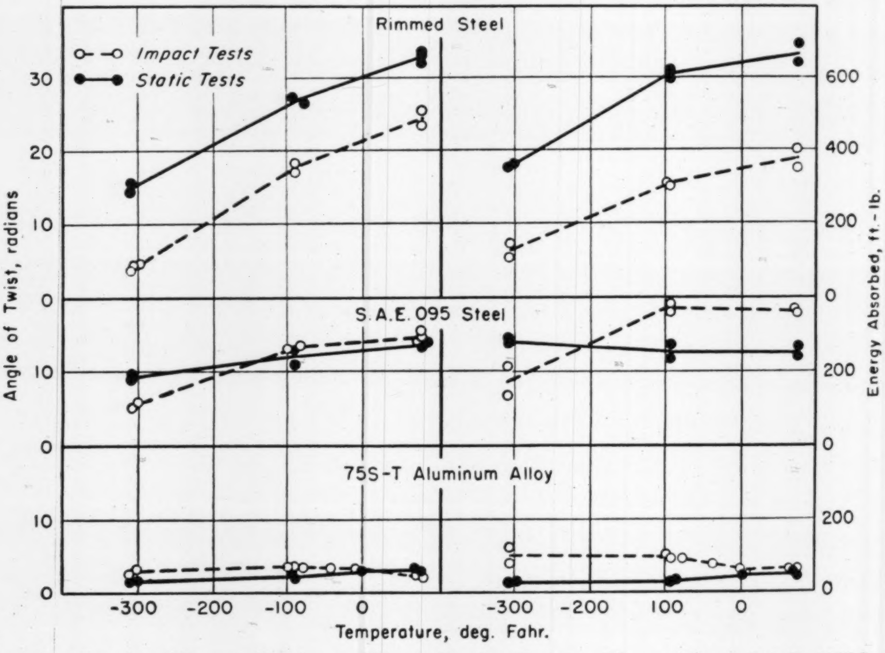


FIG. 10.—Results of Impact Torsion and Static Torsion Tests on Rimmed Steel, S.A.E. 1095 Steel, and 75S-T Aluminum Alloy.

The results of impact torsion and static torsion tests on rimmed steel, S.A.E. 1095 steel, and 75S-T aluminum alloy are shown in Fig. 10. Since the state of stress in torsion is such that the maximum shearing stress is equal to the maximum principal stress, it is to be expected that the temperature of transition from a ductile to a brittle fracture will be higher than that for the unnotched tension specimen of the same material as shown in Fig. 5. All of the torsion tests resulted in ductile fractures, although the temperature necessary to produce brittle fracture in torsion impact tests of the rimmed steel is apparently only slightly less than −310 F. The results on the rimmed steel show that the increased rate of strain in the torsion impact tests at the temperatures used has caused a considerable decrease in ductility (as measured by total angle of twist) and in total energy absorbed by the specimen. The S.A.E. 1095 steel appears to be slightly more ductile and absorbs more energy in the impact test at room temperature than in the static test at room temperature. At low temperatures, however, the reverse is true. Data obtained by Greene and Stout (**11**) on the effect of speed of straining on the ductility at room temperatures, of specimens of hardened 1.10 per cent carbon tool steel, show that the ductility increases with rate of strain. The data on S.A.E. 1095 steel are consistent with those of Greene and Stout at room temperature.

The aluminum alloy appears to have greater ductility and absorbs more energy in impact than in static tests when tested at low temperatures.

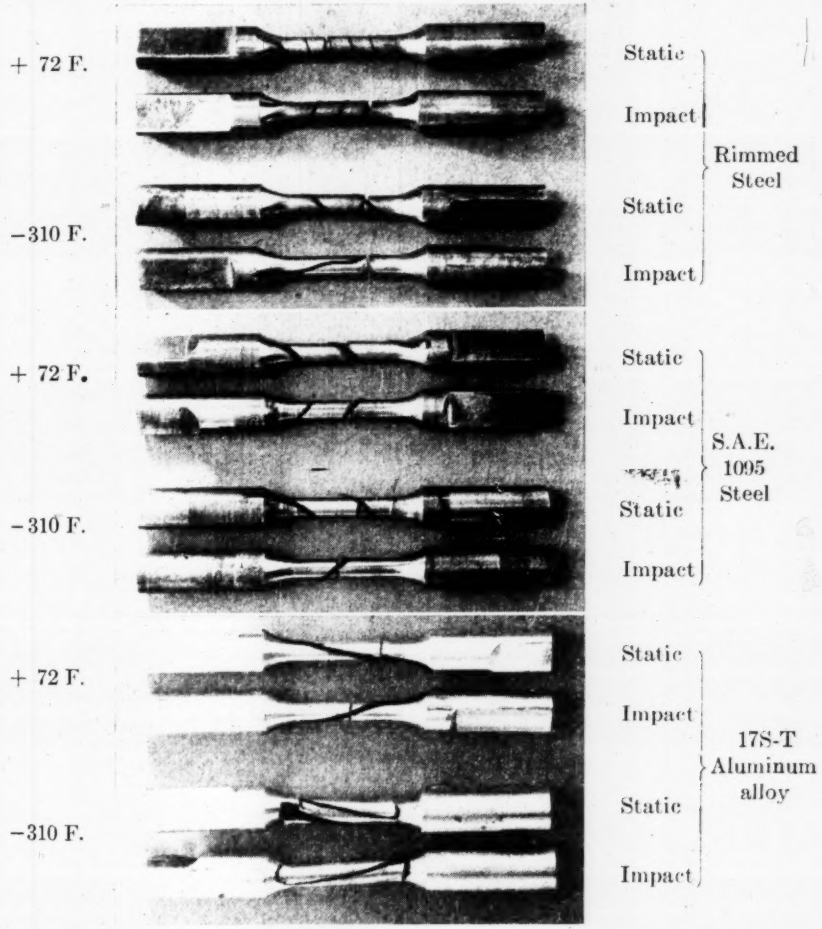

+ 72 F.
−310 F.
+ 72 F.
−310 F.
+ 72 F.
−310 F.
Static
Impact
Static
Impact
Rimmed Steel
Static
Impact
Static
Impact
S.A.E. 1095 Steel
Static
Impact
Static
Impact
17S-T Aluminum alloy

FIG. 11.—Fractured Torsion Specimens.

Figure 11 shows several typical fractured specimens tested in torsion. The high ductility of the rimmed steel tested in static torsion at +72 F. is evident from the spiral line which was a straight line before the specimen was tested. The rimmed steel tested at −310 F. in impact shows high localized yielding, but the total angle of twist is much less than for the specimen tested under static loads at room temperatures. The 75S-T aluminum alloy tested at room temperature shows the familiar transverse fracture, but the specimens tested at −310 F. fractured in longitudinal shear. The change from a transverse fracture to a longitudinal fracture occurs in the impact tests at a temperature near −90 F. Tests made at this temperature fractured on a transverse plane, but longitudinal cracks occurred in the specimen. Specimens tested at −100 F. and less fractured on a longitudinal plane. This type of fracture in the impact tests probably introduced some error in measurement of angle of twist and energy absorbed since the parts of the specimen rubbed together after fracture occurred. The change from a transverse fracture to a longitudinal fracture occurs in the static tests at a temperature less then −90 F.

SUMMARY AND CONCLUSIONS

The primary purpose of this paper was to extend the work on the influence of various factors on the mode of fracture of metals to cover a wider range of the combinations of external conditions which tend to embrittle a metal. In particular, an examination was made of the mode of fracture of a metal subjected to high rates of strain at low temperatures under different states of stress. Although the paper emphasizes the effects of factors which relate to the external conditions imposed on a metal, this is not to be interpreted that internal conditions are of lesser importance. There is need for further study of the influence of factors associated with the internal state of a metal and closely connected with its life history.

Certain combinations of external conditions may cause brittle behavior of a metal which exhibits great ductility under other conditions of use. For example, low-carbon steel when subjected to a state of stress most favorable to plastic deformation (torsion) was found to exhibit a fracture approaching the brittle type when subjected to a low temperature and a high rate of strain. Until a much needed satisfactory theory is given to explain the phenomenon of fracture, the results of tests which indicate the effect of various combinations of embrittling factors must be relied upon for predicting the type of fracture that will occur under various conditions of use.

Acknowledgments:

This investigation was conducted as a part of the work of the Engineering Experiment Station of the University of Illinois. Acknowledgment is made to Prof. F. B. Seely, Head of the Department of Theoretical and Applied Mechanics, for his help and suggestions in planning the tests and in preparing this paper.

REFERENCES

(1) F. B. Seely, "The Strength Features of the Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 535 (1940).

(2) H. Neuber, "Theory of Notch Stresses: Principles for Exact Stress Calculation," David Taylor Model Basin, *Translation No.* 74 (1945).

(3) P. G. Jones and H. F. Moore, "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 610 (1940).

(4) D. S. Clark, "Influence of Impact Velocity on the Tensile Characteristics of Some Aircraft Metals and Alloys," *Technical Note No.* 868, National Advisory Committee for Aeronautics, October, 1942.

(5) R. O. Fehr and E. R. Parker, "Measurement of Dynamic Stress and Strain in Tensile Impact," *Experimental Stress Analysis*, Vol. 1, No. 1 (1943).

(6) M. J. Manjoine, "Influence of Rate of Strain and Temperature on Yield Stresses of Mild Steel," *Transactions*, Am. Soc. Mechanical Engrs., Journal of Applied Mechanics, Vol. 66, pp. A211–A218 (1944).

(7) D. J. McAdam, Jr., G. W. Geil, and R. W. Mebs, "The Effect of Combined Stresses on the Mechanical Properties of Steels Between Room Temperature and—188 C.," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 448 (1945).

(8) S. L. Hoyt, Discussion of "On the Transition from a Ductile to a Brittle Type of Fracture in Several Low-Alloy Steels," by P. G. Jones, *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 554 (1943).

(9) D. J. McAdam, Jr. and R. W. Mebs, "An Investigation of the Technical Cohesive Strength of Metals," T. P. No. 1615, *Metals Technology*, Am. Inst. Mining and Metallogical Engrs., Vol. 10, August, 1943.

(10) G. Sachs and J. Lubahn, "Effects of Notching on Strained Metals," *Iron Age*, Oct. 15, 1942, pp. 48–52.

(11) O. V. Greene and R. D. Stout, "A Study of the Influence of Speed on the Torsion Impact Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1292 (1939).

DISCUSSION

MR. A. R. BOBROWSKY.[1]—It appears that high deformation occurs in the specimens close to the point of fracture. I wonder whether a specimen might have heated rapidly at that point during straining to become somewhat warmer, so as to make it susceptible to further deformation? Were temperature measurements made during test?

MR. PAUL G. JONES (*author*).—I think it is true that the high local deformation would certainly increase the temperature of the specimen. That is not the cause of the initial localized deformation, because the deformation must come first. However it would make the metal susceptible to further deformation. I think that would also be true at the low temperatures. I do not believe the refrigerant would carry away the heat fast enough to equalize the temperature throughout the specimen. Temperature measurements were not made at points of local deformation.

---

[1] Lewis Flight Propulsion Lab., National Advisory Committee for Aeronautics, Cleveland, Ohio.

SOME ASPECTS OF THE EFFECT OF METALLURGICAL STRUCTURE ON FATIGUE STRENGTH AND NOTCH-SENSITIVITY OF STEEL*

By T. J. Dolan[1] and C. S. Yen[1]

Synopsis

A study was made to determine the extent to which certain changes in metallurgical structure (produced by different heat treatments) affected the fatigue strength and notch sensitivity of several steels, and to ascertain whether the types of metallurgical constituents that improve localized toughness in a Charpy impact test also prove beneficial in fatigue. Experimental data are presented from static, fatigue, and impact tests, on two alloy steels and one carbon steel, quenched and tempered to approximately the same hardness level. Duplicate tests were made for each steel in a drastically quenched-and-tempered condition and in a slowly quenched-and-tempered condition.

In general, the steels rapidly cooled in quenching exhibited higher yield ratios, slightly higher endurance limits, and less notch-sensitivity in fatigue than the same steels slowly quenched and tempered. The fatigue strength of tempered martensitic specimens with a 60-deg. V-notch, ranged from 11 to 44 per cent higher than for the same metals heat treated to a structure of pearlite plus ferrite (but having the same hardness and tensile strength). Essential differences are pointed out in the structural actions occurring in a fatigue test as compared with those in the single-blow notched-bar impact test. As a result of the tests it was concluded that no direct functional relationship was apparent between the notch-sensitivity in a fatigue test and that in a Charpy test.

Several investigators (**1**, **2**, **3**)[2] have indicated that rapidly quenched steels having a structure of tempered martensite exhibited greater toughness (as measured by energy absorption in Charpy bending impact tests) than specimens containing tempered pearlite or various mixtures of other high-temperature transformation products. These observations, however, are usually based on the lower transition temperature (and increased energy values above this temperature) for the tempered martensitic steel.[3]

There is always a tendency to apply or extrapolate this type of data to other conditions of service in actual engineering structures which do not involve the sudden rupture of a notched bar under a single heavy blow; it is doubtful whether the engineering qualities desirable for application to other operating conditions can be adequately estimated on the basis of a Charpy impact test. For instance, there is no definite reason to believe that the fatigue strength of a member subjected to repeated loading has a direct relationship to the impact energy absorption exhibited by a small notched specimen. One might speculate that for fatigue tests of a notched member at a high stress level, a single-blow, notched-bar impact test might represent a limiting condition approached by a fatigue test in which only a few cycles of stress were involved before fracture. However, at a lower stress level (involving a very large number of repetitions of stress) the phenomena that govern the fracture are somewhat different and hence cannot be adequately appraised on the basis of comparative energy absorbing capacity when suddenly ruptured in a Charpy test.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Research Professor of Theoretical and Applied Mechanics and Research Graduate Assistant, respectively, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 689.

[3] As the temperature of the test is decreased, a certain "transition temperature" range is reached, above which the metal exhibits a rather large energy absorbing capacity in the Charpy test and below which a pronounced drop in energy absorbing capacity is exhibited.

Nevertheless, the question arises as to whether the beneficial constituents in the metallurgical structure that improve the localized toughness in a notched-bar impact test will also prove of benefit in retarding or resisting the initiation and propagation of fracture in a member subjected to a large number of cycles of stress. In other words, is the difference in metallographic structure (such as free ferrite or pearlite *versus* tempered martensite) a primary factor in determining the fatigue life of a member at a high stress level or in influencing the endurance limit of a material at a very large number of cycles of stress? In general, mixed structures often are obtained in the heat treatment of commercial products, and small isolated volumes of weaker constituents might influence the fatigue strength.

The phenomena observed and the evidence presented as "notch-sensitivity" in an impact test are widely different from those of a fatigue test. Therefore, it would be desirable to know whether the notch-sensitivity of a metal in a fatigue test is related in any manner to the notch-sensitivity as determined from a Charpy impact test, and furthermore, to learn whether changes in metallurgical structure that affect the sensitivity in an impact test have a corresponding influence on the fatigue properties.

Object and Scope of Investigation

An investigation was made to study the extent to which apparent differences in microstructure of steel affected the fatigue properties and Charpy impact values obtained for several steels heat-treated to the same hardness. For this purpose a relatively low hardness of approximately 101 Rockwell B scale was selected to enable the production of quite different metallographic structures having the same final hardness. Determinations were made of static tensile properties, hardness, Charpy impact values, and flexural fatigue properties of both notched and unnotched specimens; at this hardness level, seven groups of specimens were tested whose hardness ranged from a maximum of 102 Rockwell B scale to a minimum of 99, averaging approximately 101.2. These consisted of one plain carbon steel and two alloy steels, each heat treated by two different methods to produce two different types of metallurgical structure.

For steel of a higher hardness level (above the hardness range of pearlite plus ferrite resulting from slow quenching), two additional groups of specimens were prepared, one treated by oil quenching and tempering, and the other transformed by quenching in an isothermal bath (austempering) to a hardness of 27 Rockwell C scale. These two series were planned to enable comparison of a bainitic structure with a tempered martensite, from the viewpoint of fatigue strength and fatigue notch sensitivity.

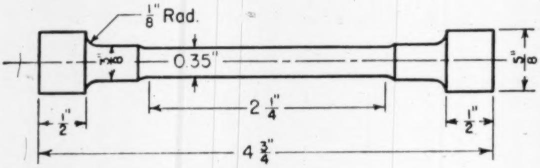
In the single-blow notched-bar impact test, the laboratory studies were conducted over a wide range of temperatures, including the critical temperature on all of the alloy steels in the conditions as mentioned above.

MATERIALS, SPECIMENS, AND METHOD OF TESTING

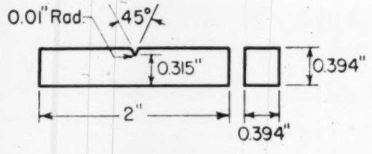
All of the steels were received in the form of hot-rolled, round bars having the chemical compositions shown in Table I. The $\frac{3}{4}$-in. diameter bars of S.A.E. 1045 and the $\frac{7}{8}$-in. diameter bars of S.A.E. 3140 and 2340 were cut into segments $9\frac{3}{8}$ in. long and normalized at 1550 F. These pieces were then turned in a lathe to $\frac{5}{8}$-in. or $\frac{1}{2}$-in. diameter preparatory to quenching and drawing. For the S.A.E. 1045 and 3140 steels, each segment $9\frac{3}{8}$ in. long by $\frac{5}{8}$ in. diameter was utilized to make two Charpy impact and one tension test specimen; the $\frac{1}{2}$-in. diameter segments were each used to make three flexural fatigue test specimens. The dimensional details of these specimens are indicated in Fig. 1. For the tests of S.A.E. 2340 steel, it was found that the $\frac{5}{8}$-in. diameter was not large enough to cause measureable hardness gradient across the section after

TABLE I.—CHEMICAL ANALYSES OF STEELS TESTED.

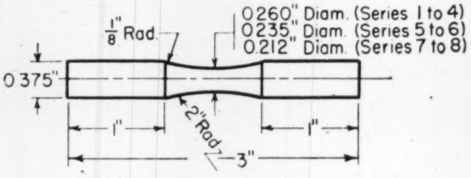
S.A.E. Steel	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent
No. 1045........	0.44	0.75	0.014	0.021	0.21	...	...
No. 3140........	0.37	0.75	0.017	0.030	0.178	1.33	0.65
No. 2340........	0.40	0.74	0.019	0.020	0.28	3.48	...


$\frac{1}{8}$" Rad.
$\frac{3}{8}$"
0.35"
$\frac{5}{8}$"
$\frac{1}{2}$"
2 $\frac{1}{4}$"
$\frac{1}{2}$"
4 $\frac{3}{4}$"

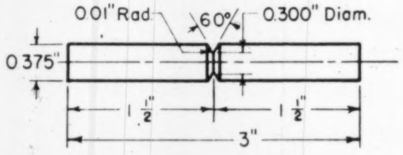
(a) Static Tension


0.01" Rad.
45°
0.315"
0.394"
2"
0.394"

(b) Charpy Impact Bending


$\frac{1}{8}$" Rad.
0.260" Diam. (Series 1 to 4)
0.235" Diam. (Series 5 to 6)
0.212" Diam. (Series 7 to 8)
0.375"
2" Rad.
1"
1"
3"

(c) Unnotched Fatigue


0.01" Rad.
60°
0.300" Diam.
0.375"
1 $\frac{1}{2}$"
1 $\frac{1}{2}$"
3"

(d) Notched Fatigue

FIG. 1.—Details of Test Specimens.

quenching, and this size was used in roughing out for heat treatment all the specimens of S.A.E. 2340 that were tested at the lower hardness level.

Preliminary exploratory tests were first made with several quenching treatments and various drawing temperatures in order to select those treatments giving the desired structures for each material and the proper values of hardness.

TABLE II.—HEAT TREATMENT AND STATIC MECHANICAL PROPERTIES FOR EACH GROUP OF SPECIMENS.

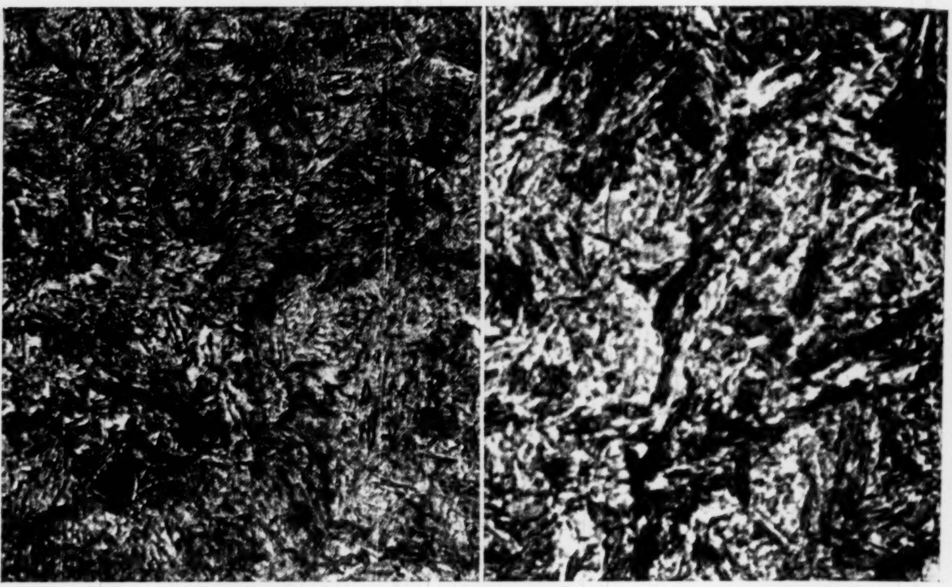
S.A.E. Steel (1)	Series (2)	Austenitize ½ hr., deg. Fahr. (3)	Quenched In (4)	Temperature 1 hr. deg. Fahr. (5)	Rockwell Hardness (6)	Elongation in 2 in. per cent[a] (7)	Reduction of Area, per cent (8)	Yield Strength (0.2 per cent offset), psi. (9)	Tensile Strength, psi. (10)	Yield Ratio: Col. (9) / Col. (10) (11)
No. 1045...	1 2	1520 1520	Water Oil	1210 1050	B99.7 B102.3	22.5 21.3	62.8 57.3	83 800 81 900	104 700 119 900	0.801 0.683
	Ratio:		Value for Series 1 / Value for Series 2		0.97	1.06	1.10	1.02	0.87	1.17
No. 3140...	3 4	1520 1520	Oil Air Blast	1300 1050	B101.3 B99.4	25.5 24.7	60.7 68.0	87 200 74 600	108 200 109 000	0.807 0.680
	Ratio:		Value for Series 3 / Value for Series 4		1.02	1.03	0.89	1.17	0.99	1.19
No. 2340...	5 6	1450 1450	Oil Air Blast	1200 None	B101.9 B102.3	23.7 19.5	67.0 43.5	96 000 75 500	115 700 122 100	0.830 0.618
	Ratio:		Value for Series 5 / Value for Series 6		0.99	1.22	1.54	1.27	0.95	1.34
	6T	1450	Air Blast	700	B101.2	22.3	58.0	78 800	118 900	0.663
	Ratio:		Value for Series 5 / Value for Series 6T		1.01	1.06	1.16	1.22	0.97	1.25
No. 2340...	7 8	1450 1450	Oil Lead at 755 F.	1100 None	C26.9 C27.0	19.8 24.2	63.3 64.4	117 600 106 500	130 100 134 200	0.904 0.794
	Ratio:		Value for Series 7 / Value for Series 8		0.99	0.82	0.98	1.10	0.97	1.14

[a] For specimens of type in Fig. 1(*a*); 2 in. gage length = 6 diameters.

NOTE: Odd numbered series (1, 3, 5, 7) will be referred to as rapidly quenched; even numbered series (2, 4, 6, 6T) are relatively slowly quenched. Series 8 was transformed isothermally in quenching.

Table II lists the final heat treatments utilized, and the resulting static mechanical properties obtained for each series of samples. Those series desgnated by odd numbers (1, 3, 5, 7) were considered to be rapidly quenched, whereas those of even numbers (2, 4, 6, 6T) were relatively slowly cooled; series 8 comprised the specimens transformed isothermally. One group of specimens (series 6) consisted of S.A.E. 2340 that had been slowly quenched only, (without subsequent tempering). For comparison with this group, a second series 6T was tested of the same steel slowly quenched, but subsequently tempered at a low temperature (700 F).

The tests to compare oil-quenched-and-tempered *versus* austempered S.A.E. 2340 steel were made at a higher hardness level (approximately 27 Rockwell C scale), but the hardness was kept low enough to allow final machining of the specimens to remove any decarburization or warping caused by the heat treatment. After normalizing, these specimens for the tests of series 7 and 8 were rough machined slightly oversize prior to heat treatment. Only sufficient stock was left on the diameters (approximately 0.040 in. for the specimen in Fig. 1(*c*), to allow for final truing-up in the finishing operations and for the removal of decarburized surface layers.



(*a*) Series 3, S.A.E. 3140 steel, oil quenched from 1520 F. (× 500). Etched in nital. (Note.—The "as quenched" structures for the S.A.E. 2340 steel in Series 5 and 7 were practically identical with the above in appearance.)

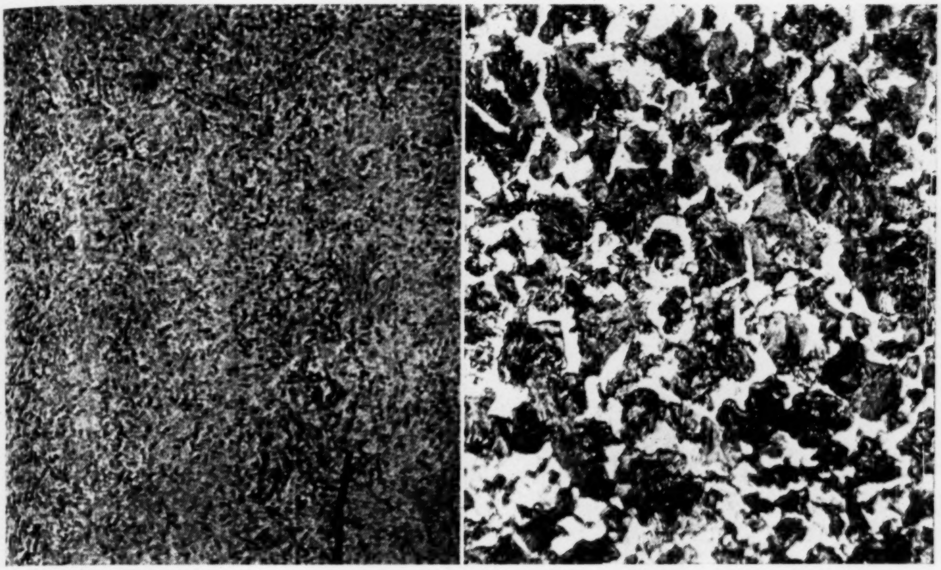
(*b*) Series 1, S.A.E. 1045 steel, water quenched from 1520 F. (× 2000). Etched in nital. (The microstructure is primarily martensite with no retained austenite, but includes some small islands of nodular pearlite indicated by the dark patches.)

FIG. 2.—Photomicrographs of "As Quenched" Structures.

(*a*) Series 1, Water quenched from 1520 F; tempered at 1210 F. (Structure is mainly tempered martensite; finely dispersed carbides partially coalesced or spheroidized. Some traces of nodular pearlite existed.)

(*b*) Series 2, oil quenched from 1520 F; tempered at 1050 F. (Mixed structure principally pearlite plus ferrite with traces of tempered martensite.)

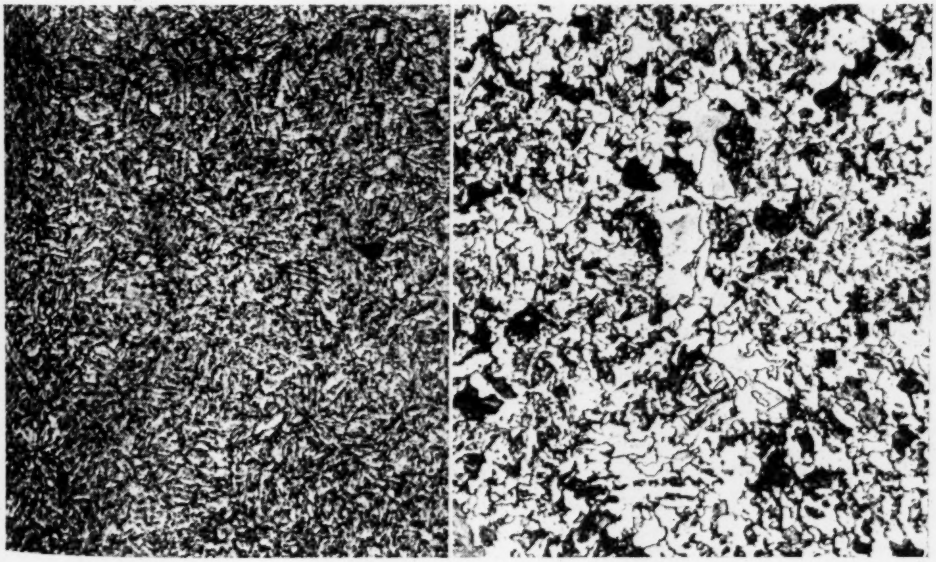
FIG. 3.—Micrographs of S.A.E. 1045 Steel, Etched in Picral (× 500).



(*a*) Series 3, oil quenched from 1520 F.; tempered at 1300 F. (The structure is essentially tempered martensite with no apparent traces of retained austenite or of higher temperature transformation products.

(*b*) Series 4, Air Blast Quenched from 1520 F.; tempered at 1050 F. (The structure is primarily pearlite with ferrite outlining colonies of pearlite.)

FIG. 4.—Micrographs of S.A.E. 3140 Steel, Etched in Picral (× 500).



(*a*) Series 5, Oil Quenched from 1450 F.; tempered at 1200 F. (The structure is mainly tempered martensite with no traces of retained austenite or of higher temperature transformation products.)

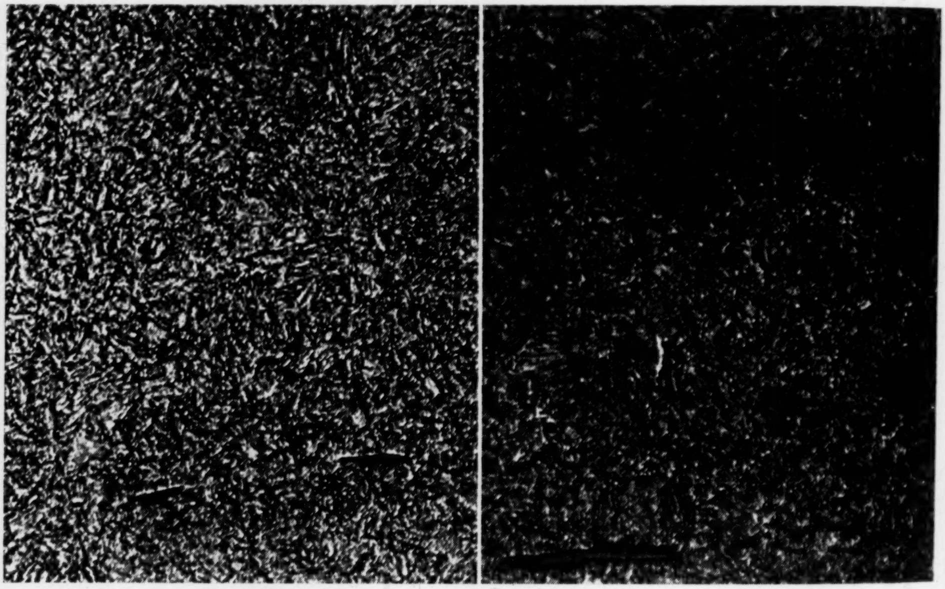
(*b*) Series 6T, Air Blast Quenched from 1450 F.; tempered at 700 F. (The structure is a mixture of ferrite and fine pearlite with some traces of carbides coalesced on tempering. The microstructure for Series 6 was almost identical with that shown above for Series 6T.)

FIG. 5.—Micrograph of S.A.E. 2340 Steel, Etched in Nital (× 500).

A preliminary study was made to determine the optimum time of the isothermal quench by holding small samples for different periods of time up to 60 min. at the predetermined temperature (755 F.). Hardness readings obtained on each sample indicated that the transformation was practically complete in about 15 min. The treatment selected for series 8 consisted of austenitizing at 1450 F. followed by quenching in an isothermal lead bath at 755 F. for 25 min. before removing and air cooling.

The photomicrograph in Fig. 2(*a*) has been included to show the appearance of the S.A.E. 3140 steel as oil quenched (series 3) prior to tempering. Similar micrographs of the S.A.E. 2340 steel in the "as quenched" condition for series 5 and 7 showed their structures to be almost identical in appearance with Fig. 2(*a*). Examination of a sample of the water-quenched S.A.E. 1045 steel in the" as quenched" condition indicated isolated islands of a darker etching constituent (Fig. 2(*b*)), which at high magnification appeared to be nodular pearlite.

Sample photomicrographs of each of the materials in the conditions as tested are shown in Figs. 3 to 6, inclusive. In these illustrations, it will be observed that those materials which were rapidly quenched exhibited final structures consisting primarily of tempered martensite (Figs. 3(*a*), 4(*a*), 5(*a*), 6(*a*)) with few traces of higher temperature transformation products or of retained austenite. Those series that were relatively slowly quenched had a final structure consisting mainly of grains of free ferrite and fine lammelar pearlite (Figs. 3(*b*), 4(*b*), 5(*b*).) The austenitic grain size of each of these materials was relatively



(*a*) Series 7, oil quenched from 1450 F.; tempered at 1100 F. (The structure consists of tempered martensite with finely dispersed cementite particles that are not completely resolved at this magnification.)

(*b*) Series 8, Quenched From 1450 F. in Isothermal Bath at 755 F. (The structure is coarse bainite. The precipitated carbide particles appear to have agglomerated in lamellae in the matrix.)

FIG. 6.—Micrograph of S.A.E. 2340 Steel at Higher Hardness Level of 27 Rockwell C scale, Etched in Picral (× 600).

fine, ranging from A.S.T.M. size 6 to 8 or smaller.

The Charpy bending impact tests were made on a machine of 223 ft. lb. capacity and over a range of temperatures from −212 F. to 220 F. The static tension tests were made in an Amsler Universal Testing Machine having a capacity of 50,000 lb., and an extensometer having a 2-in. gage length was used to measure the strains. This is a non-standard gage length for tension specimens of the dimensions in Fig. 1(*a*), since the gage length is about 6 diameters instead of the more commonly employed value of $L = 4D$. However, a suitable gage of shorter length was not available, and it was deemed advisable to maintain the diameters of the tension specimens at approximately the same small size as the fatigue and impact specimens to avoid variations in properties due to mass effects in heat treating.

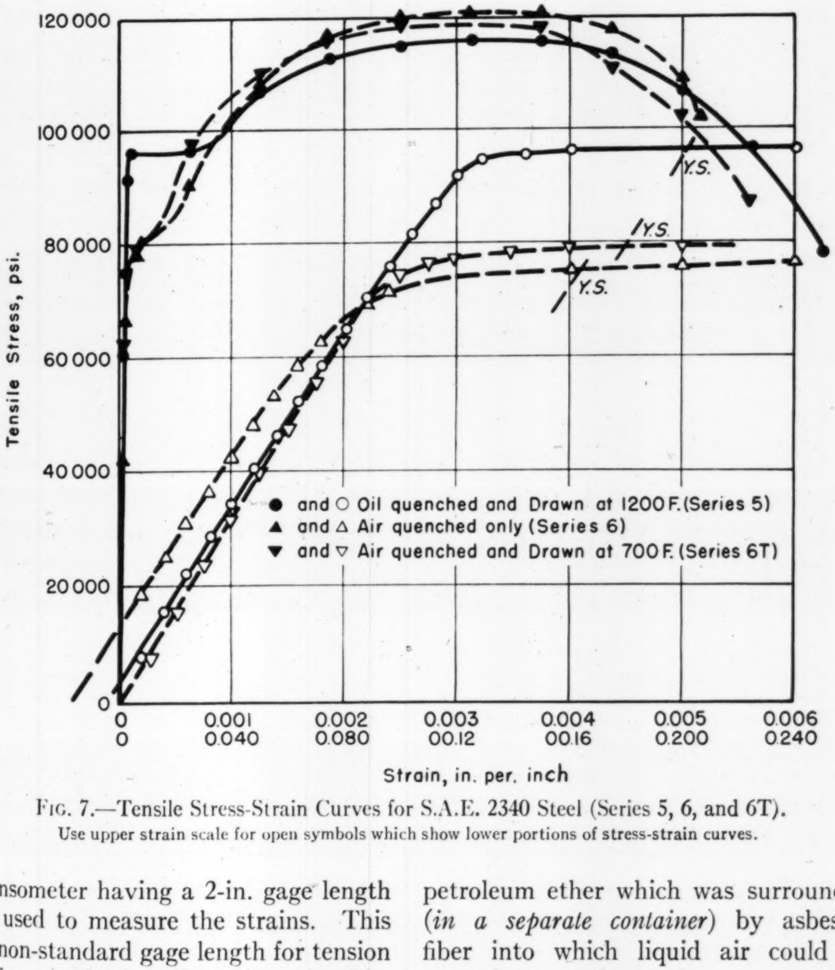


Fig. 7.—Tensile Stress-Strain Curves for S.A.E. 2340 Steel (Series 5, 6, and 6T).
Use upper strain scale for open symbols which show lower portions of stress-strain curves.

For Charpy tests at temperatures down to −110 F. an acetone bath cooled by dissolving dry ice was found satisfactory; at very low temperatures, the specimens were cooled in a bath of petroleum ether which was surrounded (*in a separate container*) by asbestos fiber into which liquid air could be poured to reduce the temperature. Great care was taken to prevent mixing of petroleum products or other oxidizable materials with the liquid air, since such mixtures become highly inflammable or create a severe explosion hazard. A few specimens were tested at tempera-

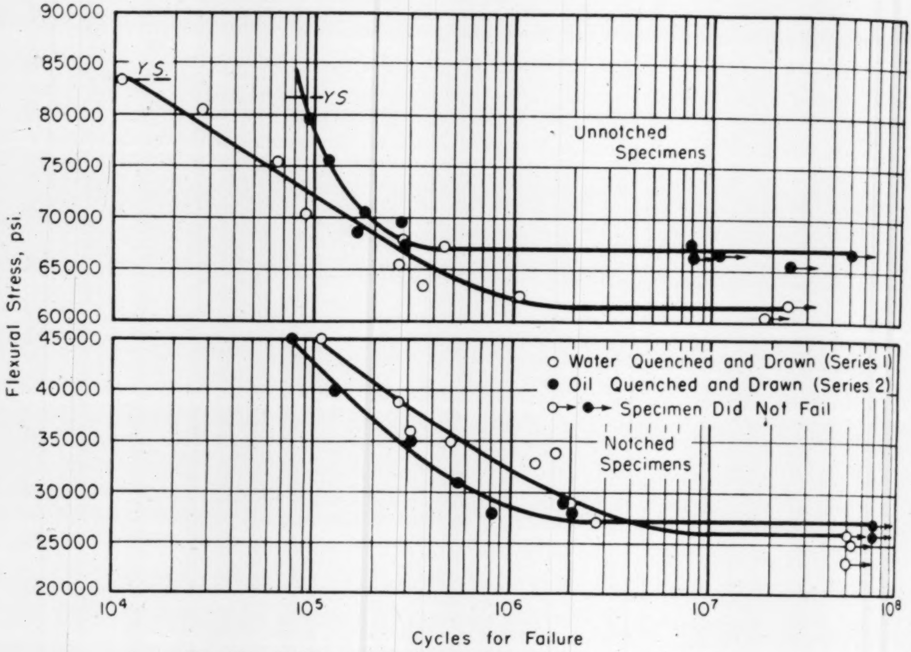


FIG. 8.—*S-N* Curves for S.A.E. 1045 Steel (Series 1 and 2).

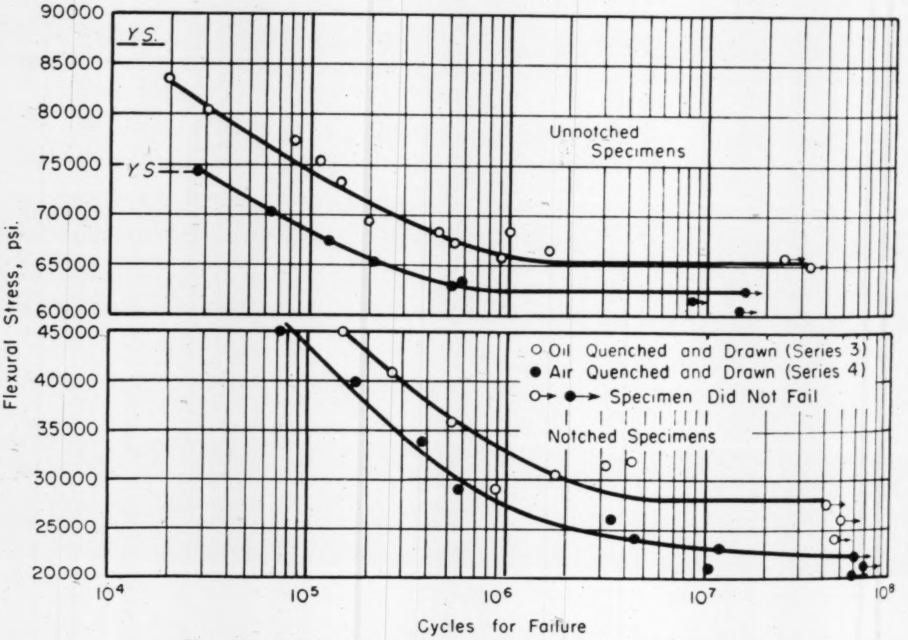


FIG. 9.—*S-N* Curves for S.A.E. 3140 Steel (Series 3 and 4).

tures above that of room temperature by heating them in an oil bath to the testing temperature. It was assumed that only a negligible change in temperature

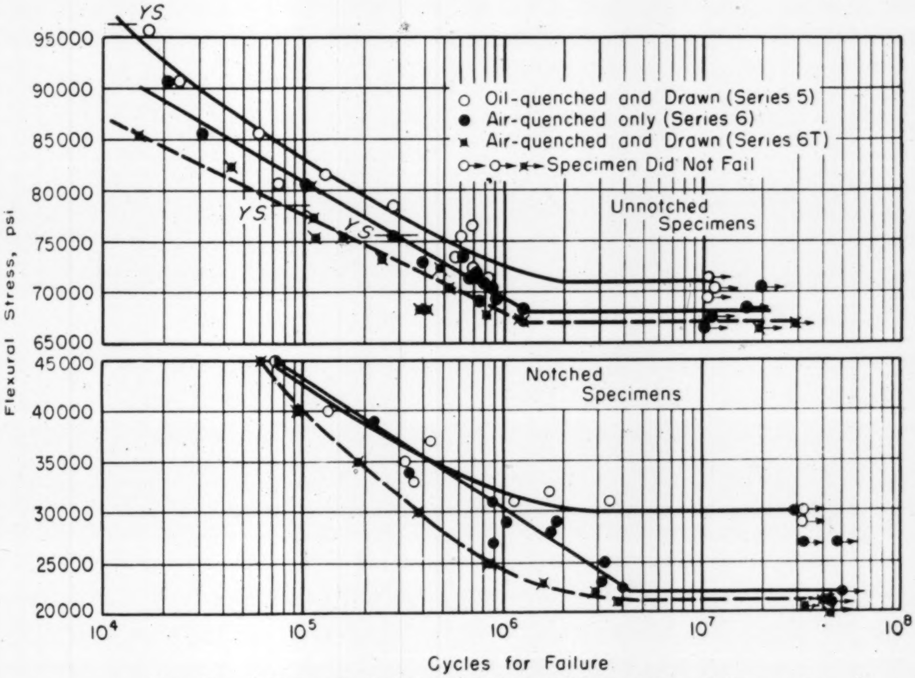


FIG. 10.—*S-N* Curves for S.A.E. 2340 Steel (Series 5, 6, and 6T).

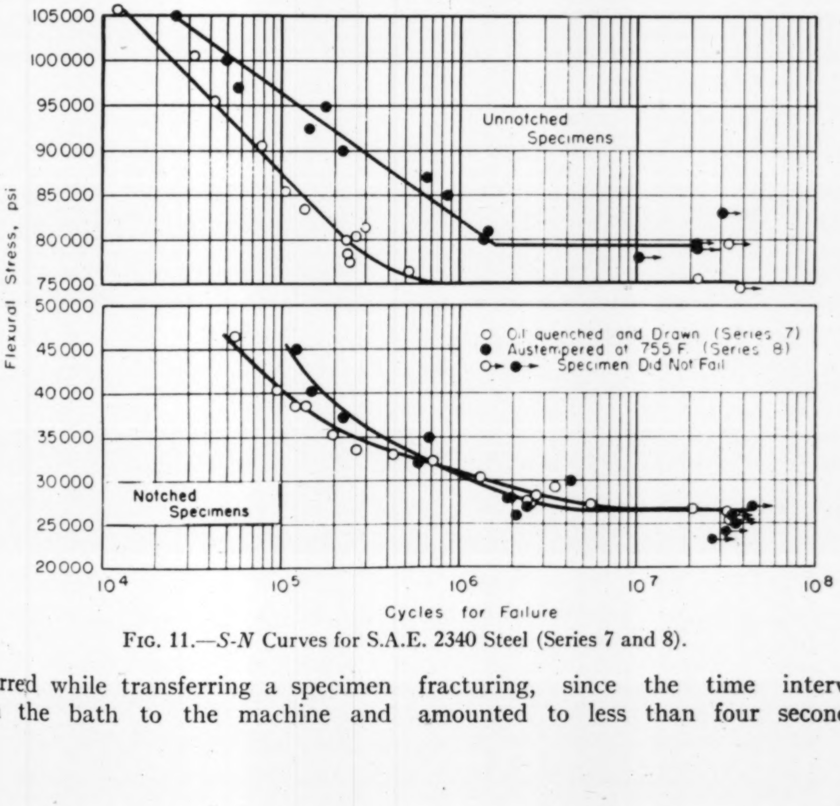


FIG. 11.—*S-N* Curves for S.A.E. 2340 Steel (Series 7 and 8).

occurred while transferring a specimen from the bath to the machine and fracturing, since the time interval amounted to less than four seconds.

Flexural fatigue tests were made of small unnotched polished specimens of the type shown in Fig. 1(*c*) and on notched specimens of the type shown in Fig. 1(*d*). In the preparation of these specimens, light finishing cuts were taken with a sharp tool in a lathe. The unnotched specimens were given a standard fatigue finish by polishing with three successively finer grades of emery paper finishing with No. 00 paper in such a manner that the scratches from each new grade of paper crossed those of the previous grade, and the final polishing scratches were parallel to the longitudinal axis of the specimen. The 60-deg. V-notch in the notched specimens was machined with a carefully ground and honed tool that had previously been examined at 50 diameters magnification in an optical projection machine for accuracy of contour. No subsequent polishing operations were performed on the notched specimen; the test surface was that left by the machining operation and was found to be free from tears or deep scratches. The diameters at the root of the notch were measured in a special fixture employing a microscope with Filar eyepiece as a reference axis and a traveling stage carrying the specimen. Movement of the stage (across the diameter of the specimen) was determined by means of an Ames dial reading to 0.0005 in.; several diameters were measured for each specimen, estimating the nearest tenth of a division.

TABLE III.—SUMMARY OF RESULTS OF IMPACT AND FATIGUE TESTS.

S.A.E. Steel	Series	Charpy Impact Tests			Endurance Limit, psi.		K^a = Ratio: $\frac{\text{Col. (6)}}{\text{Col. (7)}}$	Endurance Ratio: $\frac{\text{End. Lim.}}{\text{Ten. Str.}}$		Notch Sensitivity Index[b] $\frac{K-1}{K_t-1} = q$
		Values, ft-lb.		Trans. Temp. deg. Fahr.	Un-notched	Notched		Un-notched	Notched	
		at 70 deg. Fahr.	at −42 deg. Fahr.							
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
No. 1045....	1	28.7	8.2	>+70	61 500	26 000	2.37	0.59	0.25	0.72
	2	17.4	5.2	>+70	67 000	27 000	2.48	0.56	0.22	0.79
	Ratio: $\frac{\text{Ser. 1}}{\text{Ser. 2}}$	1.65	1.6		0.92	0.96	0.96	1.05	1.11	0.91
No. 3140....	3	120.2	108.2	−45	65 500	28 000	2.34	0.605	0.26	0.71
	4	81.9	17.7	+65	62 500	22 000	2.84	0.57	0.20	0.97
	Ratio: $\frac{\text{Ser. 3}}{\text{Ser. 4}}$	1.47	6.12		1.05	1.27	0.82	1.06	1.30	0.73
No. 2340....	5	92.5	83.6	−95	71 000	30 000	2.37	0.61	0.26	0.72
	6	31.0	11.7	+82	68 000	22 000	3.09	0.56	0.18	1.23
	Ratio: $\frac{\text{Ser. 5}}{\text{Ser. 6}}$	2.98	7.15		1.04	1.36	0.767	1.09	1.44	0.59
	6T	60.1	17.3	+65	67 000	21 000	3.19	0.56	0.18	1.15
	Ratio: $\frac{\text{Ser. 5}}{\text{Ser. 6T}}$	1.54	4.83		1.06	1.43	0.74	1.09	1.44	0.63
No. 2340	7	80.8	69.3	−120	75 000	26 500	2.83	0.58	0.20	0.96
	8	90.2	18.8	+27	79 500	26 500	3.00	0.59	0.19	1.05
	Ratio: $\frac{\text{Ser. 7}}{\text{Ser. 8}}$	0.90	3.69		0.94	1.00	0.94	0.98	1.05	0.91

[a] K = Strength Reduction Factor = $\frac{\text{Endurance limit of unnotched specimen}}{\text{Endurance limit of notched specimen}}$.

[b] In this relation, K_t = theoretical stress concentration factor.

NOTE: Series 1, 3, 5, 7 were rapidly cooled in quenching. Series 2, 4, 6, 6T were relatively slowly cooled in quenching. Series 8 was quenched in an isothermal bath at 755 F.

All flexural fatigue tests of both notched and unnotched specimens were conducted in two Krouse rotating cantilever beam machines. Each of these machines was calibrated in terms of moment developed in a dummy specimen on which was mounted two SR-4 type electric resistance strain gages. These gages served to measure the bending moment transmitted by the machine in terms of a previous calibration of the dummy specimen with dead weights. The speed of rotation of each specimen in the fatigue tests was kept between 6000 and 8000 rpm.

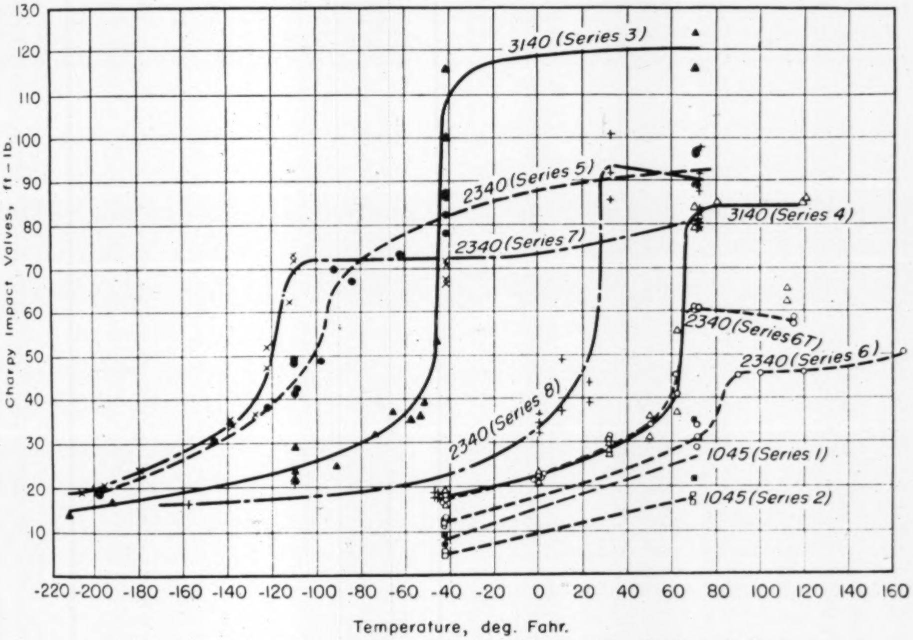


FIG. 12.—Effect of Temperature on Energy Observed in the Charpy Test.

RESULTS OF TESTS

In Fig. 7 are shown representative samples of the static tensile stress-strain diagrams for the S.A.E. 2340 steel in three test conditions. A summary of the static tensile properties obtained from curves of this type is given in Table II. The flexural fatigue test data obtained from each group of specimens are shown in the *S-N* curves of Figs. 8 to 11, inclusive, in which the ordinates represent the nominal flexural stress computed from the formula $S = \frac{Mc}{I}$ without regard for the stress concentration introduced in the notched specimens by the abrupt change in section. The short dashed lines indicated by the letters *Y.S.*, in Figs. 8 to 10, have been added to indicate the yield strength level for each of the series. (The flexure formula does not express true values of stress above these levels.)

The results of the majority of the fatigue and impact tests are summarized in Table III. In order to facilitate the comparisons of properties in both Tables II and III, there have also been included a number of ratios of each value of mechanical property for the rapidly quenched steels (series 1, 3, 5, 7) to the corresponding value of mechanical property for the slowly quenched steels (series, 2, 4, 6, 6T). Each value of hardness shown in these tables represents the average of about 18 readings, and the values shown in Table III for impact energy absorbed are the average of two to four tests in each case.

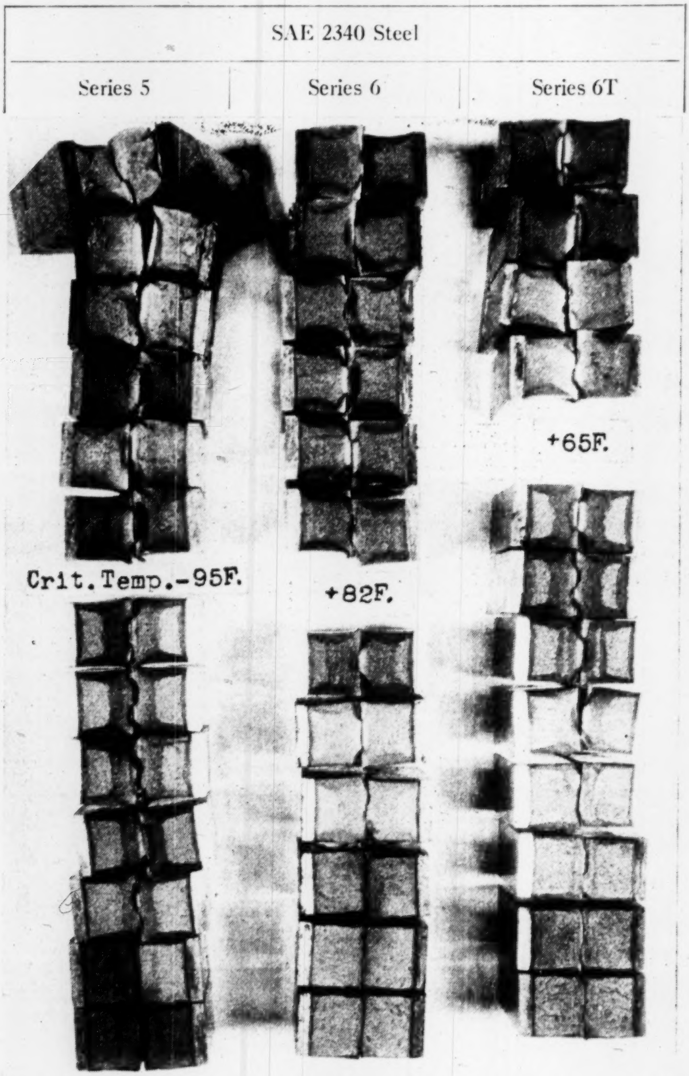


FIG. 13.—Photographs Showing Types of Fracture in Charpy Impact Specimens.

Further impact tests conducted over a wide range of temperatures are shown in the data plotted in Fig. 12. In this figure, each individual specimen has been plotted as a separate point, and rough curves have been sketched in to indicate the approximate trends of the data with a sudden transition or abrupt drop in a rather narrow temperature range. It will be noted that below the transition temperature all of the alloy steels exhibited rather low energy absorption in the Charpy tests but relatively high absorption at temperatures above this critical zone.

As a further possibility of correlation in the study of notch sensitivity, a "sensitivity index" discussed by Thum and Buchmann (**5**) and by Peterson (**4**) was calculated for each condition and listed in the last column of Table III. For the 60-deg. V-notch employed in the fatigue specimens, the stress concentration factor K_t has been determined to be 2.9 from the theoretical analysis of Neuber (**6**). Peterson has shown that the sensitivity index, *q*, is not a unique property of a material (it may be affected by a number of factors such as the sharpness of the notch), but it does serve as a rough inverse measure of the relative resistance of different groups of specimens to the damaging effects of a particular stress raiser.

Photographs of the fractured surfaces of representative Charpy impact specimens are shown in Fig. 13 to illustrate the transitions obtained from ductile fibrous fractures to partially and nearly completely brittle crystalline fractures as the temperature was lowered in each test. In general, the transition temperature was rather clearly defined by the appearance of the surfaces of the fractured impact specimens.

DISCUSSION OF RESULTS

Static Tensile Characteristics:

It is interesting to note from the data in Table II that at approximately the same hardness level the yield ratios for the slowly quenched (pearlitic) series were somewhat lower (values ranged from 0.62 to 0.68) than the yield ratios for the rapidly quenched series (for which the values ranged from 0.80 to 0.83). For the higher hardness level (Rockwell C 27) the steel transformed isothermally (series 8) had a lower yield ratio of 0.79 as compared with a yield ratio of about 0.90 for the corresponding oil-quenched-and-drawn S.A.E. 2340 steel (series 7). These results are in agreement with the data reported by Herres and Jones (**3**) which indicate that steels initially fully hardened to martensite in quenching exhibit higher yield ratios than those slowly quenched.

For S.A.E. 1045 steel, the tensile strength of the slowly quenched series 2 was 119,900 psi. as compared with only 104,700 psi. for the rapidly quenched series 1, which latter group also had a slightly lower hardness. For the other pairs of materials, however, the slowly quenched and drastically quenched steels in each case had approximately the same strength; thus the oil quenched and drawn S.A.E. 1045 steel specimens should perhaps have been drawn at a slightly higher temperature to be comparable with the other groups on the basis of hardness. However, this difference is of some interest in interpreting whether the slight increase in hardness had more influence on the fatigue strength than the widely different metallurgical structure of a companion group of specimens of the same steel.

As far as the static tests are concerned, no definite trends or tendencies may be noted of the effect of structure on either the percentage elongation or the percentage reduction of area, and the higher yield points for the rapidly quenched groups was the only quality markedly affected by the differences in methods of heat treatment. In some instances (for example the S.A.E. 1045 and S.A.E. 2340 steels), the drastically quenched steels appeared to have slightly higher ductility as represented by these two properties, but this was partially due to their slightly lower tensile strengths as compared with the slowly quenched and drawn series of the same compositions.

Impact Values:

The marked differences in Charpy impact values listed in Table III are in line with the results of other investigators. The values tabulated (for series 2, 4, 6, 6T) for tests at −42 F. are indicative of the relatively small Charpy values for pearlitic or ferritic steels at low temperatures. However, the rapidly quenched *alloy* steels (series 3, 5, 7) exhibited only a small drop in energy absorption corresponding to this decrease in temperature (from room temperature down to −42 F.); as indicated in Fig. 12, the transition temperatures for series 3, 5, 7 were at or below −42 F. The low Charpy values obtained for the water-quenched S.A.E. 1045 steel (series 1) might be a further indication that this shallow hardening steel was not completely martensitic before tempering, even though drastically quenched. The data in Fig. 12 indicate that the transition temperature for series 1 was somewhat above room temperature. These differences from the behavior of the other rapidly quenched steels could be caused by the presence of a mixed structure containing small amounts of a higher temperature transformation product as indicated by the evidence in Fig. 2(*b*).

The results of the Charpy impact tests over a range of temperatures (shown in Fig. 12) emphasize two important facts: (*a*) the transition temperatures for these steels in the various conditions as tested scattered over a wide range from −120 F. to values above room temperature, and (*b*) it is obvious that comparative results of energy absorption at a given temperature are meaningless, even for steels at the same hardness level, if some of the metals have transition temperatures in the region of the test temperature. Thus it is difficult to draw conclusions regarding "notch-sensitivity" from Charpy impact tests. It will be noted that the highest energy absorption obtained was for the S.A.E. 3140 steel (series 3) whereas the lowest transition temperature that was obtained (−120 F.) ocurred for the S.A.E. 2340 steel (series 7). At temperatures somewhat below the critical temperature for each metal, there is little significant difference in the Charpy impact values for the various steels and conditions tested. Above the transition temperature ranges there are rather marked differences in the energy absorbing capacity of the different alloys in the several conditions tested.

Fatigue Properties:

(*a*) *Fatigue Strength and Fatigue Notch-Sensitivity.*—In general, the *S-N* curves of Figs. 8 to 11 indicate a trend toward slightly higher endurance limits, and somewhat longer "finite life" at a given stress level, for the rapidly quenched groups (odd numbered series) as compared with the corresponding slowly quenched groups (even numbered series). The only test series which appeared to show abnormal scatter in fatigue data was for the tests of S.A.E. 2340, air quenched only (series 6); several specimens of both notched and unnotched types ran abnormally long (18 to 50 million cycles) without failure at stresses somewhat above the endurance limits indicated in Fig. 10. However, these few abnormal specimens were tested a few months after heat treating, and it was felt that perhaps an unstable condition existed due to lack of tempering that resulted in aging effects at room temperature. For this reason, an additional group of specimens (series 6T) was also air quenched, but tempered at 700 F. The fatigue data in Fig. 10 for series 6T show similar trends to those for series 6; the tempering treatment apparently eliminated the abnormal scatter, but also resulted in a very slight decrease in tensile strength, hardness, and endurance limit.

The values of the endurance limits listed in columns 6 and 7 of Table III were undoubtedly influenced to some extent by the differences in the static mechanical strengths resulting from the various heat treatments. Several investigators (**7**, **8**) have shown that a fairly close relationship exists between the fatigue strength and the static tensile strength; the endurance limit for ferrous metals generally ranges from 0.4 to 0.6 of the tensile strength, as indicated by the two dotted lines plotted in Fig. 14. The reader might infer that no appreciable change in endurance limit would accompany changes in metallurgical structure if the tensile strength remained unaltered. However, the upper points in Fig. 14 represent considerably more favorable fatigue characteristics than those bordering the lower limits of the scatter band, and it would be advantageous to process a member to meet these upper limits of possible fatigue strength.

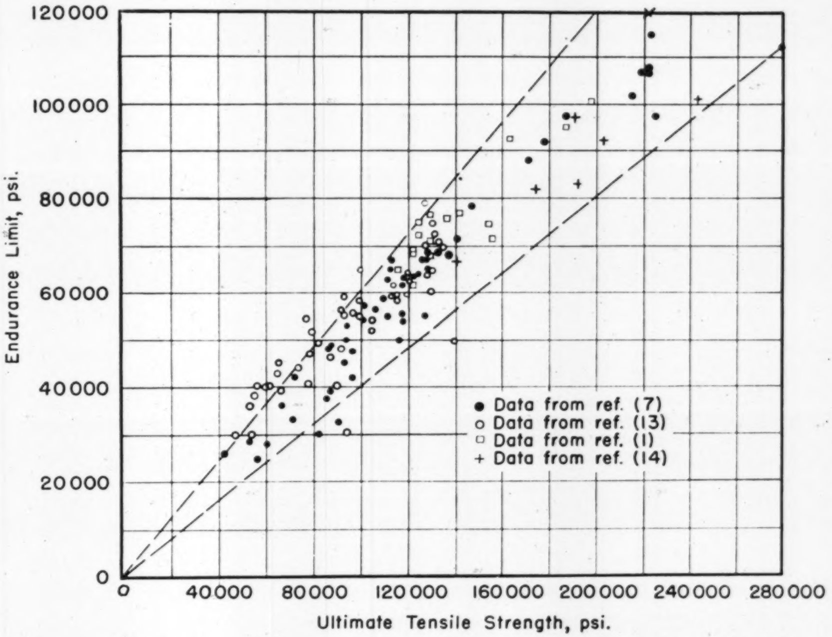


FIG. 14.—Relation Between Endurance Limit and Tensile Strength for Ferrous Metals.

Since the tensile strengths of the steels of series 1 to 6 varied slightly, the endurance ratios have been listed in columns 9 and 10 of Table III; the magnitude of this ratio may be regarded as somewhat indicative of the relative efficiency of the treatment in developing desirable fatigue characteristics. In order to facilitate comparisons of the data of Tables II and III, the principal results have been plotted in the form of a bar chart in Fig. 15. In this figure the shaded blocks represent values from rapidly quenched steels and the open blocks the more slowly quenched series; values for a given property are shown by

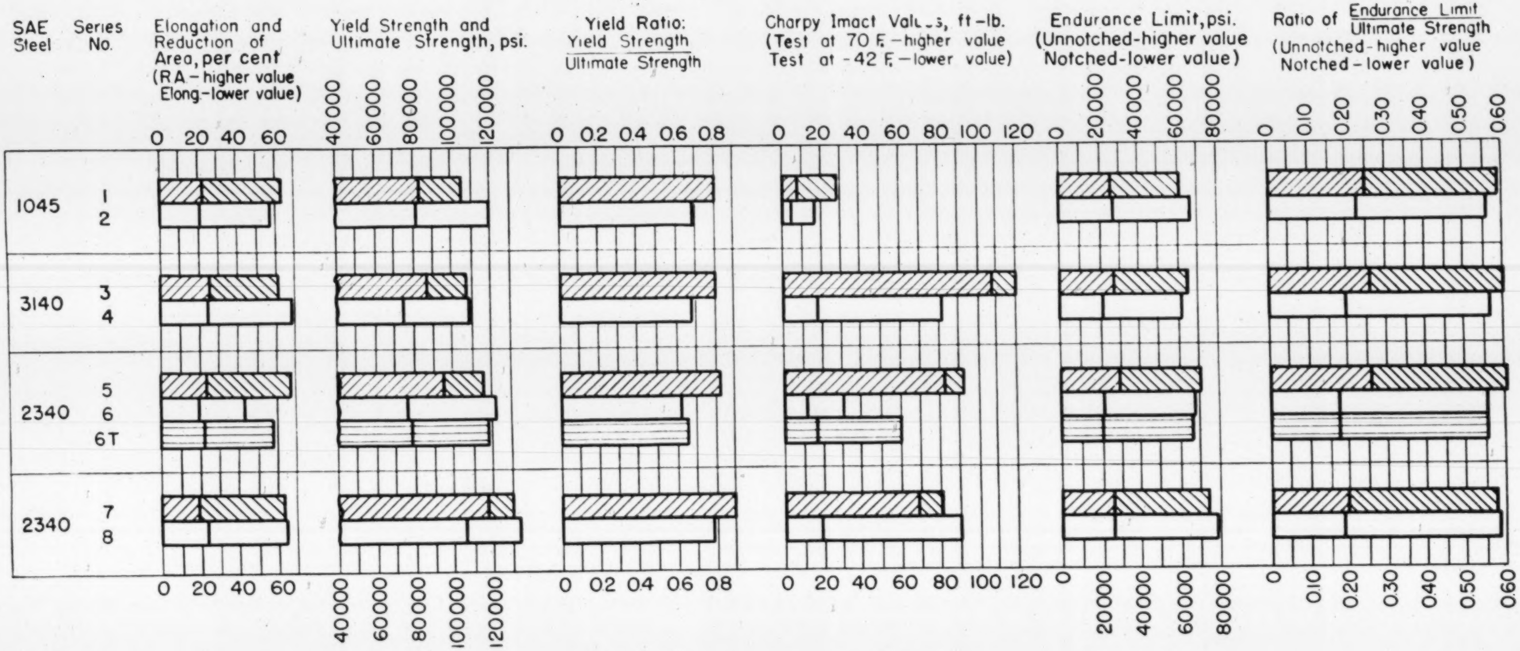


FIG. 15.—Summary of Results of Tests.

the relative lengths of the bars in any single column. It will be noted that the steels rapidly quenched had slightly higher endurance ratios (values of 0.59 to 0.61), as compared with the same steels slowly quenched (having values 0.56 and 0.57). The actual numerical differences, however, between the endurance limits of companion sets of *unnotched* specimens are relatively small. A similar increase in fatigue strength could have been produced by lowering the tempering temperature (and thus increasing the hardness and tensile strength) for the slowly quenched steels.

The differences in behavior caused by the presence of the localized stress resulting from a 60-deg. V-notch were somewhat more pronounced. The strength reduction factors, *K*, (column 8 of Table III) for rapidly quenched groups varied from only 2.34 to 2.37, whereas the slowly quenched groups exhibited higher values of *K* ranging from 2.48 to 3.19. Thus on this basis, the steels having structures of pearlite plus ferrite (slowly quenched) exhibited a somewhat greater fatigue notch-sensitivity than those having structures mainly of tempered martensite. Similarly, the "notch-sensitivity index," listed in column 11 was in each case greater for the steel in the slowly quenched condition.

It has sometimes been felt that steels with a relatively low yield ratio, or those containing pearlite + ferrite, might have more tendency to yield locally and redistribute the peak stress over a greater volume in the region of the stress raiser (and thus result in lower fatigue notch-sensitivity) than in the case of steels having structures with finely dispersed carbides and exhibiting somewhat higher yield strengths. However, the data in Table III show that the steels consisting essentially of pearlite + ferrite (and which exhibited the lower yield ratios) were considerably more notch-sensitive than those with fairly uniform dispersal of carbides (rapidly quenched) as judged by either the strength reduction factor, *K*, or the sensitivity index, *q*. In general, the rapidly quenched steels with the higher yield ratios also displayed the highest ratio of endurance limit to tensile strength as shown in Fig. 15. However, for the conditions investigated, the relationship between increase in endurance limit and yield point was apparently only qualitative in nature; plots of endurance limit *versus* yield point showed appreciable scatter, and a simple straight-line relation such as that shown by Hollomon and co-workers (1) did not adequately represent the variations in data.

(*b*) *Fatigue Properties for the Same Tensile Strength.*—The ratios of the values obtained from companion groups of specimens (as shown in Table III) may be used as one means of comparing the relative merits of the mechanical characteristics of the structures resulting from the heat treatments employed. For instance, the ratios for $\frac{\text{Series 1}}{\text{Series 2}}$ in columns 6 and 7 indicate that the actual endurance limits of unnotched and notched specimens of the rapidly quenched group were 0.92 and 0.96, respectively, of the corresponding endurance limits for the slowly quenched steel. However, it must be kept in mind in this comparison that the specimens of series 1 were not of as high a hardness and had a tensile strength only 0.87 of that for series 2 (see Table II).

It is suggested that a more equitable comparison of merit may be made by converting these actual values to a common tensile strength, or what is somewhat simpler, by expressing them in terms of tensile strength as has been done in columns 9 and 10 of Table III. On this basis, one finds that the unnotched specimens of the rapidly quenched series 1 had an endurace limit ratio 1.05 times that for the slowly quenched series 2, or equivalent to a 5 per cent increase in fatigue strength for *the same* tensile strength level. Similarly, the notched specimens of series 1 exhibited an 11 per cent increase in fatigue strength over series 2 when compared on the basis of uniform tensile strength. The trends for the S.A.E. 3140 steel (series 3 and 4, which had practically identical tensile strength) are perhaps somewhat more reassuring since the actual endurance limits and endurance ratios were both greater for the rapidly quenched series 3. Even though the rapidly quenched S.A.E. 2340 steel (series 5) had a slightly lower tensile strength than the two slowly quenched series, the fatigue properties were in each case superior to those of series 6 and 6T. There appears to be little difference in the fatigue characteristics introduced by tempering the slowly quenched S.A.E. 2340; the endurance limits for series 6 and 6T are practically identical when one considers the slight reduction in tensile strength and hardness resulting in the tempered group.

In general (excluding series 7 and 8), *the endurance ratios for unnotched specimens of the rapidly quenched groups were consistently from 5 to 9 per cent greater, and for the notched specimens were from 11 to 44 per cent greater, than the corresponding endurance ratios for the slowly quenched steels.* These comparisons may be readily visualized in Fig. 15 by observing the relative lengths of the bars plotted for the endurance ratios (last column on the right).

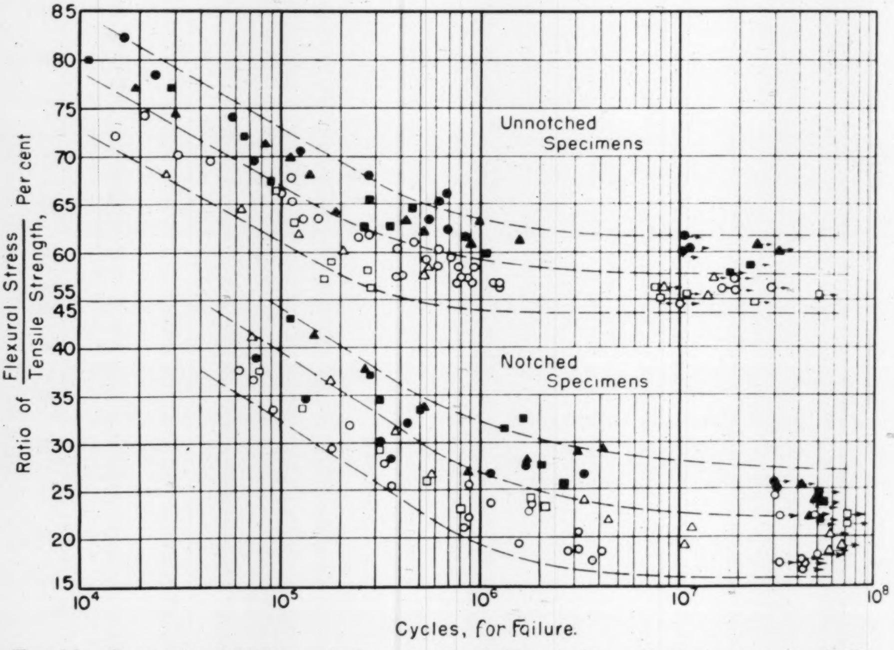


FIG. 16.—Composite *S-N* Curves in Terms of Static Tensile Strength (Series 1 to 6 and 6T).

The general trends outlined in the above discussion are further illustrated by the composite *S-N* diagrams shown in Fig. 16. In this diagram all of the points plotted in Figs. 8 to 10 have been replotted with ordinates shown as a percentage of the respective static tensile strengths for the specimens of series 1 to 6 and 6T. The same symbols for each series have been used as those employed in plotting Fig. 12; that is, the open symbols represent those groups of specimens slowly quenched, whereas the solid or closed symbols have been employed to show data for the groups that were rapidly cooled in quenching. It is obvious that these differences in heat treatment produced a definite change in fatigue properties both for notched and for unnotched specimens, as shown by the two sets of "scatter bands" that have been roughly outlined by dotted lines. In each case the upper scatter band (representing a longer "finite" fatigue life and a higher endurance limit) encloses the majority of the solid symbols, whereas the lower scatter band includes practically all of the open symbols which show data for the slowly quenched steels. Therefore, *for a given static tensile strength,* a definite trend toward increased fatigue life and fatigue strength was obtained for these three steels when heat treated to a structure consisting primarily of tempered martensite; heat treatments resulting mainly in higher temperature transformation products of pearlite plus ferrite were inferior both for polished specimens and for specimens containing a severe stress raiser.

(*c*) *Comparison of Austempered with Quenched-and-Tempered Steel.*—The data for the S.A.E. 2340 steel tested at the higher hardness level of Rockwell C 27 (in series 7 and 8) show only small differences in fatigue characteristics. In static tension, the specimens transformed isothermally in quenching (series 8) had a slightly higher ultimate strength and ductility, but a 10 per cent lower yield point than those oil quenched. The Charpy impact values for series 8 were higher at room temperature, but the material also had a higher transition temperature than series 7. The only appreciable difference in fatigue properties between these two groups is noted in the greater strength reduction factor of 3.00 (and the greater notch sensitivity index) for series 8, as compared with 2.83 for series 7. (See Table III, columns 8 and 11). The austempered specimens had a slightly higher fatigue strength in conventional tests of polished specimens but a slightly lower fatigue strength for notched specimens, than the comparison group of specimens oil quenched and drawn to the same hardness.

In a somewhat similar series of tests reported by Aral (9) at a higher hardness level of 46 Rockwell C, only negligible differences were noted in the fatigue strengths of conventional polished specimens. Tests of N.E. 8742 steel after three different heat treatments (namely: austemper, martemper and temper, or oil quench and temper) all gave endurance limits of approximately 122,000 psi. which was about 55 per cent of the tensile strength. In tests of S.A.E. 1095 steel at a still greater hardness level of 54 Rockwell C, Carreker (10) obtained the following endurance limits for polished specimens:

Heat Treatment	Endurance Limit, psi.
Water quench and temper at 550 F	124 000
Austemper, 550 F	130 000
Martemper and temper at 550 F	160 000

At this high hardness level, however, the drastic quenching treatment results in a brittle material, and the high residual stresses induced in quenching are not entirely alleviated by the tempering treatments. It is felt that the differences in endurance limit noted by Carreker may be partially attributed to the differences in inherent residual stresses rather than due to the alterations in metallographic structure resulting from the heat treatments. By martempering the bar is allowed to come to a fairly uniform temperature tending to minimize the stress gradient throughout the piece during the transformation. The greater temperature gradient induced by more drastic treatments, such as water quenching, may result in extremely high residual stresses due to the non-uniform volume expansions and contractions arising from the formation and subsequent transformation of martensite. Almen (9) has emphasized the importance of residual stresses in affecting the fatigue strength of a metal since these stresses are largely additive to those nominally computed stresses imposed by the external loads.

In this connection, it should be pointed out that in the tests of series 1 to 6 and 6T, the drastically quenched steels required a higher tempering temperature in each case than those that were slowly quenched, in order to draw them back to approximately the same hardness. Thus, even though the more drastically quenched steels probably had higher inherent stresses prior to tempering, there is a possibility that the higher tempering temperatures relieved the micro residual stresses to a slightly greater extent than is the case for the slowly quenched steels that were tempered at lower temperatures. A portion of the improved fatigue properties of the tempered martensitic groups might thus be partially due to a greater freedom from undesirable types of inherent stress.

(*d*) *Explanation for Influence of Metallographic Structure.*—Aside from the influence of residual stresses, however, a previously proposed theory of fatigue (12) offers a possible explanation why the rapidly quenched steels in the present tests exhibited better fatigue properties than the same steels with pearlitic structures. The submicroscopic actions that progress prior to the initiation of a fatigue crack are greatly affected by the interactions between atoms in the unordered regions existing along crystalline boundaries and the distorted slip planes and layers produced by crystal fragmentation in previous stress cycles. For materials having a structure containing very finely dispersed hardening constituents (such as the precipitated carbides in a tempered martensitic structure) there would exist maximum amounts of unordered material (bounding the individual crystalline domains) at the initiation of a test. For structures with larger sized crystalline groups of atoms (such as the larger plates of ferrite and cementite in a pearlitic steel) there would be a smaller proportion of the total volume contained in the (unordered) intercrystalline regions. Hence, the first stage of progressive damage, which consists primarily of slip and crystal fragmentation, would be materially retarded because of the restraints imposed by the unordered material and because of the small "free-lengths" over which gliding along slip planes could occur without intersecting a neighboring crystal of different orientation or composition. Conversely, pearlitic structures consist of relatively larger agglomerations of ferrite and of cementite, and hence are more favorably grouped for slip and crystal fragmentation to proceed. Evidence of this increased restraint of initial slip is shown by the higher yield strengths of the tempered martensitic steels. Thus, the statistical average behavior of a metal with finely dispersed hardening constituents would, in general, be expected to show improved fatigue life and a higher fatigue strength than a material that appears to be coarse-grained or to have larger agglomerations of individual crystalline constituents.

COMPARISON OF FATIGUE AND IMPACT PROPERTIES

Lack of Correlation Between Fatigue and Charpy Notch-Sensitivity:

Except for tests of series 1, all specimens having structures primarily of tempered martensite exhibited superior Charpy impact properties (lower transition temperatures and higher energy absorption) than were exhibited by the *same steels* slowly quenched. Also it has been observed that the fatigue characteristics as judged by the endurance limit and the strength reduction factor, were somewhat improved when a given steel was more rapidly quenched in heat treating. On this basis it might arbitrarily be inferred that a direct correlation exists between the behavior measured in the Charpy test and that evaluated by a fatigue test. *However, any such comparison should be regarded as valid only for the same steel treated to the same tensile strength*, and the correlation is purely *qualitative only;* it is partially accidental and not susceptible to a direct mathematical or quantitative evaluation.

As evidence of the lack of correlation between fatigue and Charpy notch-sensitivity, Fig. 12 indicates that the specimens of series 2 exhibited the poorest impact properties (greatest notch-sensitivity) of any of the materials tested. However, the data plotted in the last two columns of Fig. 15, indicate that the fatigue notch-sensitivity of this group was smaller (higher endurance limit of notched specimens) than for series 4, 6, or 6T, which latter groups exhibited lower impact notch-sensitivity. Similarly, the specimens of series 1 exhibited very poor impact properties, whereas the fatigue notch-sensitivity was smaller than for series 4, 6, or 6T, and its general resistance to the reduction in strength due to the introduction of a notch in the fatigue specimens was practically as good as that shown for series 3 and 5, which groups exhibited the best resistance in the impact test (lowest impact notch-sensitivity). The reader will note that for the rapidly quenched steels (of series 1, 3, 5) there was a wide divergence in Charpy impact values and transition temperatures, but little or no change in endurance ratio or fatigue notch-sensitivty. Again, the slowly quenched series 4, 6, and 6T exhibited little difference in fatigue properties but showed appreciable differences in energy absorption in the Charpy test. Thus, even for specimens at the same strength level, a comparison of the varous steels tested, indicates that *there is not a direct qualitative relationship between notch-sensitivity as evidenced in the fatigue test with that determined from a Charpy impact test.*

It is probably true that a method of processing a steel that results in improved impact properties might also result in some improvement of other load resisting properties as measured by certain methods of testing. However, the degree to which this improvement in other properties becomes evident may vary widely for alloys of altered composition, for other methods of loading (static or repeated), or for states of strain and strain rates somewhat different from those developed in a Charpy bar. Under a single blow which leads to fracture, a rather complex state of stress is developed and is accompanied by a relatively high rate of strain over a localized region at the root of the notch, which leads to sub-microscopic reactions or behavior quite different from that which occurs during the repetitions and reversal of load in a fatigue test.

Contrasts in the Mechanical Actions Contributing to Failure:

In this section a brief discussion is presented outlining some of the differences in the mechanical interactions that occur on the atomic scale, together with differences in strain rate on a macro scale, that occur in a Charpy test as contrasted with a fatigue test. In each of these tests the significant action which leads to failure originates in a more localized region than can satisfactorily be treated by means of the concepts commonly used by engineers in analyzing large scale (phenomenological) behavior. Hence, an approach has been made (12) based on studies in the physics of matter and utilizing the differences in behavior observed for atoms grouped in an ordered lattice (crystalline) as compared with those of unordered or imperfect lattice arrangement (amorphorus). On an atomic scale, the interactions between ordered regions (crystallites) and the unordered material surrounding the crystalline domains, result in the formation of atomic bonds of high energy content. Under the influence of external loads, the increase in bond energy between atoms leads to a forced relaxation (and redistribution of bond energy) or to disruption of the bond.

The short time involved in the Charpy test does not permit any extensive spontaneous relaxation of high-energy bonds between atoms to take place, and hence the possible beneficial readjustments between atoms existing in the partially unordered regions along the crystal boundaries is suppressed. The primary actions therefore consist of bond disruption and crystal fragmentation. Conversely, two types of relaxation of high energy bonds are possible during the repeated stressing in a fatigue test: (*a*) *damaging* forced relaxation during loading, and (*b*) *beneficial* spontaneous relaxation. Furthermore, the disruption of bonds in a fatigue test (that finally leads to formation of a crack) progresses as a phenomenon dependent upon load intensity and number of cycles of loading; it is governed by the probability of a simultaneous occurrence of sufficient thermal energy and of load energy to rupture a given bond in any individual cycle. The process of bond rupture is thus governed by chance and may be expressed in general form by statistics; the accumulation of progressive damage leading to the formation of a crack depends upon the *number* of repetitions of load. It is difficult to see how this cycle dependent process can be related in any direct manner to energy absorption of an impact specimen fractured with a single blow.

For the same reason there seems to be no direct correlation between the basic structural actions that occur (in the fatigue test as compared with the impact test) when notched specimen are considered, and hence the quality called "notch-sensitivity" which is glibly discussed in connection with both types of test, is probably a composite measure of different phenomena in the two cases. "Notch-sensitivity" is a vague concept evolved by engineers to express in a qualitative manner the lack of resistance of a material to the development and spread of a crack at the root of a "stress raiser" such as a notch. For a fatigue test it is usually measured in terms of the reduction in strength of a notched specimen as compared with the endurance limit of unnotched specimens. Quantitative relations, such as the sensitivity index, have been formulated by relating this strength reduction to the theoretical stress concentration factor obtained from some type of mathematical analysis.

From a pedagogical viewpoint, attempts by engineers to obtain a mathematical expression for notch-sensitivity in terms of a "theoretical" stress concentration factor are futile; the analyses on which the theoretical factors are based depend upon the assumptions of an ideal isotropic material which is perfectly elastic and homogenous and whose strength properties are not influenced by previous stress history or by the time during which loads are applied. Actually, however, when dealing with the localized stresses developed in small volumes of polycrystalline aggregates one must realize that the individual crystals are basically anisotropic, that the metal is far from homogenous, and that elastic action is a myth; localized microscopic or submicroscopic inelastic readjustments occur in the material at relatively low stresses. Furthermore, these readjustments (inelastic behavior) are to some extent time sensitive, particularly for those metals whose melting points are not too high above the temperature of testing (such as lead, tin, etc., at room temperature).

The concepts of notch-sensitivity in an impact test are even more indefinite than those for a fatigue test, since the term as applied to a Charpy test must involve the relative energy absorption capacity over a range of temperatures, as well as a knowledge of the transition temperature. The fact that the Charpy test is much more sensitive to the effects of varying temperature or to changes in velocity of impact (than is a fatigue test in terms of altered temperatures or frequency of stressing) is further evidence of the lack of a definite functional relationship between notch-sensitivity in the two types of test. It should be pointed out in this respect that in the minute volumes of material subjected to structural damage, there is wide divergence in the strain rates encountered: extremely high rates of strain in localized regions are developed during the process of fracture of a Charpy bar. Conversely, in a fatigue test, which is usually conducted with sinusoidally varying stresses, the strain rate is a maximum at the time the stress is zero, and decreases to a zero strain rate at the instant the stress reaches a maximum amplitude. The differences in the effect of these two types of strain history on the mechanical strength developed by the material are obvious. The behavior of a fatigue specimen tested at a frequency of 100,000 cycles per minute would not be expected to be comparable with the structural actions that occur in an impact specimen fractured in 1/100,000 of a minute.

In considering the actions occurring around stress raisers, the convenient assumptions of statistically isotropic and statistically homogenous material must be abandoned; the phenomena governing the failure occur at a much lower level of association than that of the usual polycrystalline aggregate subjected to a test of a phenomenological (large-scale engineering testing) nature. The interactions between and within crystals and the interactions between individual atoms (together with the alterations introduced by the parameters of time, temperature, and previous strain history) lead to markedly different behavior from that which might be predicted on the basis of elastic theory; furthermore, consideration of the effects of these parameters leads to the generalization that there is no basic reason to expect a direct correlation between notch-sensitivity as observed in an impact test and notch-sensitivity as measured in a fatigue test.

Conclusions

It is realized that there are many variables of a metallurgical nature that have not been investigated in these tests. However, within the scope of the materials and heat treatments employed, the following conclusions seem justified:

1. In general, the rapidly quenched

groups of alloy steels (series 3 and 5) exhibited impact properties superior to those for the steels slowly quenched (series 4, 6, and 6T), as evidenced by higher energy absorption and much lower transition temperatures, in the Charpy impact test. The austempered samples (series 8) had an intermediate transition temperature when compared with the tempered pearlitic and martensitic steels; S.A.E. 1045 steel exhibited poor Charpy properties whether rapidly or slowly cooled in quenching (series 1 and 2).

2. The endurance limit for *unnotched* specimens of steels consisting primarily of tempered martensite was found to be slightly higher (*about 5 to 9 per cent greater*) than that for the same steel with a structure of pearlite plus ferrite heat treated to develop the same static tensile strength. These differences are not considered of primary importance, however, since a similar increase in fatigue strength could be obtained by lowering the tempering temperature (and thus increasing the tensile strength) for the slowly quenched steels.

3. The endurance limits for *notched* specimens of the tempered martensitic steels were definitely superior (*ranging from 11 to 44 per cent greater*) when compared with those for the same steels heat treated to a structure of pearlite plus ferrite having the same tensile strength.

4. Austempered S.A.E. 2340 steel (series 8) had a slightly higher endurance limit for unnotched specimens, but also exhibited slightly greater fatigue notch-sensitivity, than the same steel oil quenched and tempered to the same hardness of 27 Rockwell C scale (series 7).

5. The fatigue notch-sensitivity of the slowly quenched steels (series 2, 4, 6, and 6T) was in each case greater (greater strength reduction caused by a stress-raiser) than for the same steel rapidly cooled in quenching (series 1, 3, 5).

6. No direct functional relationship was evident between the concepts of notch-sensitivity in a fatigue test and the notch-sensitivity evidenced in a Charpy impact test. A rough qualitative correlation was indicated for comparisons of the *same* material at the *same* hardness and tensile strength level, but even this qualitative relationship was inaccurate (and the relative order of notch-sensitivity was reversed in the two types of test) when comparing materials of different chemical analyses.

7. The Charpy test develops a higher rate of strain at peak stress (than the fatigue test), and fracture under impact is not dependent upon the cumulative chance effects developed during the repetition and reversal of load which are of paramount importance in the submicroscopic phenomena leading to failure in fatigue. Direct correlations between fatigue properties and Charpy values do not appear to be feasible.

Acknowledgments:

This investigation has been conducted as a part of the work of the Department of Theoretical and Applied Mechanics and of the Engineering Experiment Station of the University of Illinois in cooperation with the Office of Naval Research, United States Navy. Appreciation is expressed to the Office of Naval Research for its sponsorship of this project (Contract N6ori-71-T.O.IV). Special acknowledgment is made of the assistance rendered by W. J. Craig, Special Research Associate, and G. M. Sinclair, Graduate Assistant, who conducted the metallographic studies and obtained the photomicrographs. The austempering treatment was conducted with equipment in the Department of Mining and Metallurgical Engineering, and with the advice of Prof. W. H. Bruckner of that department.

REFERENCES

(1) J. H. Hollomon, L. D. Jaffe, D. E. McCarthy, and M. R. Norton, "The Effects of Microstructure on the Mechanical Properties of Steel," *Transactions*. Am. Soc. Metals, Vol. 38, pp. 807–844 (1947).

(2) P. R. Kosting, "Progressive Stress Damage," Surface Stressing of Metals, Am. Soc. Metals, pp. 143–190 (1947).

(3) S. F. Herres and A. F. Jones, "A Method for Evaluating the Toughness of Steel," *Metal Progress*, Am. Soc. Metals, September, 1946, pp. 462–469.

(4) R. E. Peterson, "Methods of Correlating Data From Fatigue Tests of Stress Concentration Specimens," in Stephen Timoshenko 60th Anniversary Volume, The Macmillan Co., New York, N. Y., pp. 179–183 (1938).

R. E. Peterson, "Relation Between Life Testing and Conventional Tests of Materials," ASTM BULLETIN, No. 133, March, 1945, p. 9.

(5) A. Thum and W. Buchmann, Dauerfestigkeit und Konstruktion, Darmstadt Technishe Hochschule, *Material Prufungsanstalt*, Heft 1, 80 pp., Mitteilugen (1932).

(6) H. Neuber, "Theory of Notch Stresses: Principles for Exact Stress Calculation," David Taylor Model Basin Translation No. 74 (1945).

(7) H. F. Moore and T. M. Jasper, "An Investigation of the Fatigue of Metals," *Bulletin No. 136*, Engineering Experiment Station, University of Illinois, (1923).

(8) D. O. Morris, "Composition and Physical Properties of Steel in Relation to Fatigue," Reprint No. 20, *Symposium on Failure of Metals by Fatigue*, University of Melbourne, December, 1946.

(9) M. M. Aral, "Endurance Limit of N. E. 8742 Steel in the Austempered, Martempered and Oil Quenched and Tempered Condition," Master of Science Deg. Thesis, Dept. of Mining and Metallergy, University of Illinois (1945).

(10) R. P. Carreker, "The Endurance Limit of S.A.E. 1095 Steel in the Austempered, Martempered, and Water Quenched and Tempered Condition," Master of Science Deg. Thesis, Dept. of Mining and Metallurgy, University of Illinois (1947).

(11) J. O. Almen, "Fatigue of Metals as Influenced by Design and Internal Stresses," Surface Stressing of Metals, Am. Soc. Metals, pp. 33–84 (1947).

(12) A. M. Freudenthal and T. J. Dolan, "The Character of Fatigue of Metals," *Fourth Progress Report*, Office of Naval Research Contract N6-ori-71-T.O.IV., University of Illinois, February, 1948.

(13) B. B. Westcott, "Fatigue and Corrosion-Fatigue of Steel," *Mechanical Engineering*, Vol. 60, November, 1938, pp. 813–822, 828.

(14) S. L. Hoyt, "Metals and Alloys Data Book," Reinhold Publishing Corp., New York, N. Y., p. 93 (1943).

DISUSSION

Mr. R. E. Peterson.[1]—In recent years there have been a number of statistical theories of fatigue strength proposed, and it may be of interest to consider what implications these theories might have with regard to quenched-and-tempered steels, as compared to unquenched steels.

It seems to me that from these theories we might expect the following three effects: First, higher notch-sensitivity for the quenched steels. That, I think, has been shown by quite a number of investigators to be the general trend. Second, we might expect less scatter. There the evidence is not so complete, and it would be interesting to have Mr. Dolan's comments. Third, we might expect less size effect, percentage-wise, with the quenched materials. There we have very little information, particularly with quenched steels. Some work has been done with unquenched steels, for instance, with large axles, but very meager information exists with regard to large quenched specimens.

I would be interested to have Mr. Dolan's comments with regard to these last two points particularly.

Mr. John M. Lessells.[2]—This paper by Mr. Dolan is of extreme interest. Any attempt so far made to correlate fatigue strength with microstructure rests with French engineers.[3]

There is one question which occurs to me. When a steel is quenched, high compressive stresses are developed in the outer layers, so in comparing fatigue and impact data obtained by test bars, presumably cut from a larger bar, it will be of importance to correlate the results with the position in the original bar. Since fully quenched steels are being dealt with, such residual stresses will be high.

Residual stresses in a quenched steel may, of course, be a combination of the macro and micro type. While it is true that the cutting up process will relieve the macro type of stress, there will be micro stresses left. Does the author believe that this factor may have contributed to the "scatter" obtained by him?

Mr. H. B. Knowlton.[4]—This paper is the most valuable contribution which we have seen concerning the relation of microstructure and static mechanical properties to fatigue strength.

We do not believe that it can be stated too strongly that all engineering steels are made to perform some useful service, and that the function of testing should be to predict the probable usefulness of parts made of different types of steel, or heat treated for different ranges of hardness and mechanical properties. Unfortunately a great deal of the published technical data does not give much information with regard to probable service performance of steel parts. There has been a tendency lately to lay a great deal of stress upon the merit of heat treating so as to produce 100 per cent martensite before tempering. Papers by Crafts and Lamont, Hollomon, and others, have shown very clearly that yield ratio and impact strength are much better if the steel is 100 per cent martensite before tempering. The conclusion is sometimes drawn that the mechanical or physical properties of steel depend entirely upon the degree of martensite present.

---

[1] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[3] R. Cazaud and L. Persoz, La Fatigue des Metaux, Dunod, Paris (1937).

[4] Supervisor of Materials Engineering, International Harvester Co., Chicago, Ill.

It must be remembered, however, that martensite is not a definite entity. It is probably more correct to speak of a martensitic type of structure. Hardness, strength and other physical properties of martensitic structures vary quite widely with carbon content.

It must also be remembered that in commercial practice there is a wide variation of hardenability from heat to heat of any given type of steel. From an engineering performance standpoint we are most vitally concerned with the minimum properties, which will be obtained with the lowest hardenability heat falling within the specified limits under which the steel was purchased. It will be a surprise to many metallurgists to learn that with a minimum hardenability lot of steel, it is impossible to obtain 100 per cent martensite in many commercial parts. With the most popular medium carbon alloy steels, such as S.A.E. 3140, 4140, 5140, and 8640, oil quenching will not produce 100 per cent martensite, even at the surface of a $\frac{1}{2}$-in. round specimen; yet all of these steels have been used very satisfactorily for many automotive parts larger than $\frac{1}{2}$ in. in diameter.

It is possible to produce 90 per cent martensite in many automotive parts by oil quenching, but in the tractor industry and some other industries where sections are large, it is not practicable to produce 90 per cent martensite in critical sections. For example, if $\frac{3}{4}$-in. radius may be selected as typical of high stressed areas, such as roots of keyways and splines, and 90 per cent martensite is considered the desirable structure, it follows that S.A.E. 5140 steel would be satisfactory for parts up to $\frac{3}{8}$ in. in diameter, and that S.A.E. 8640 and 4640 would be satisfactory up to diameters about 1.25 in. These steels are commercially used, however, for parts which are larger in diameter.

It is probably true that breakage of most moving parts of machinery is due to fatigue failures. Tension tests, impact tests, and even the determination of transition temperatures of impact tests do not yield very much information concerning fatigue strength. This paper is of particular value in that it shows the relation of some of the structures to fatigue strength. We are particularly interested to note that the reduction in fatigue strength of specimens containing pearlite and ferrite, as well as tempered martensite, was only about 5 to 9 per cent as compared with those which were entirely tempered martensite.

In many cases it would be more economical to design parts slightly larger and permit the use of low-alloy steels and structures which are not 100 per cent tempered martensite. For example, consider a shaft which is subject to bending or torsion fatigue. The strength of such a part is proportional to the cube of the diameter. An increase of the diameter of 3 per cent is equivalent to 10 per cent increase in fatigue strength.

We are also interested in the results obtained with fatigue test of notched specimens. The data indicate a much greater difference between notch fatigue strength of specimens heat treated for different percentages of martensite. There does not seem to be a great deal of published information on this subject, and the small amount of data which we have seen is not conclusive. It should be borne in mind that commercial articles, such as gears and axles, quite probably contain a considerable degree of internal stresses after heat treating and that these stresses have a pronounced effect upon the observed fatigue strength or the actual service performance of the finished parts. These internal stresses may depend upon the size and design of the finished part and may not necessarily correlate with stresses found in conventional specimens.

MR. P. R. KOSTING.[5]—There are two points I should like to bring out. One is concerned with labeling as "tempered martensite," a product which has a transition temperature as high as −40 F.

The other is that when an absolute comparison is made of the notched specimens it is disconcerting to observe that in the region of finite life a softer brittle material in the notched condition appears to have as good a life as a stronger tough material.

MR. W. E. JOMINY.[6]—I feel a bit deflated this morning to find that the metallurgist is just a step beyond the blacksmith, and I hope it isn't too true, because I think we have done quite a little bit of study of the effect of microstructure on steel properties.

This paper is a very valuable one. It attempts to answer some of the questions we have been asking for some time. I wonder, however, whether the emphasis should be put on rapid quenching and slow quenching? I think if you choose the proper steel you can cool rather slowly and still get martensite, so it seems to me the hardenability of the steel as well as cooling rate comes into the picture.

I think the emphasis should be put on microstructure, and that perhaps there is not too much difference in fatigue values between pure bainite tempered and tempered martensite.

I have one question, what size specimen was quenched? Also, I might suggest that machining off some of the surface stressed metal ought to effect the fatigue values. I think surface stresses are a rather important issue here.

MR. G. C. RIEGEL[7] *(by letter)*.—What tests can discriminate at the earliest state of fabrication between suitable and unsuitable material for an engineering structure or component?

To this question, the authors have sought an answer. Their reasoning that two modes of failure are commonly encountered in ferrous materials and represent the most troublesome engineering problems, namely, (1) the progressive separation or fatigue failure, and (2) the brittle separation or sudden fracture. Both of these failures may terminate without warning and with disastrous effects.

Their more specific question (a good one) asks if these two modes of failure are related in fundamental origin.

While they have taken two commonly used methods of mechanical testing, employing test specimens, for making an approach to this problem, namely fatigue tests and notched-bar bending tests, they report no common relationship between the results of the two types of tests.

Quite likely, material behaves in accordance with the constraints placed upon it. The lack of similarity of the modes of stressing probably accounts for the inability to find a common denominator for evaluating the two types of tests.

In other words, the kinds of stresses which reside within the crystals, their subdivisions and aggregates, the kinds of stresses which are imposed externally, all cause a variety of response which we see manifest by the influence of temperature, duration of stress, medium in which the stresses operate (corrosive or neutral), and the number of forces acting at one time.

---

[5] Metallurgical Engineer, Watertown Arsenal, Watertown, Mass.

[6] Staff Engineer, Chrysler Corp., Detroit, Mich.

[7] Chief Metallurgist, Caterpillar Tractor Co., Peoria, Ill.

Until our mechanical testing can resolve more of these forces and the magnitude of their interrelationship, the materials engineer will still have to rely upon testing full scale models, components, or materials according to the capability of the test to discriminate between those that offer the greatest resistance to the inception or propagation of a crack under a given condition of imposed stresses.

MESSRS. T. J. DOLAN AND C. S. YEN (*authors' closure*).—The authors wish to take this opportunity to thank the discussors for the interest they have shown in this paper and for emphasizing the importance of the subject. It is unfortunate that the manuscript was not preprinted since some of the questions raised regarding the size of samples treated, and the relative amounts of martensite produced in the quench, would have been made available to the discussors.

Mr. Peterson suggests that the general principles of statistical analyses would predict that quenched steels should have higher notch-sensitivity, less scatter in fatigue, and less effect of size on fatigue strength. It is true that most of the fatigue data available in the literature indicate that quenched steels exhibit greater notch-sensitivity than annealed or normalized steels, but these comparisons have generally been made on the basis of widely different strength levels for the two types of heat treatment. One of the surprising results in the present paper was that the rapidly quenched steels displayed *lower* notch-sensitivity than those slowly quenched and drawn back to the *same* hardness and tensile strength. Of course, all of these steels were quenched to some degree. However, the air-quenched series probably would not be thought of as representative of quenched steels since the structures produced contained mainly high-temperature transformation products.

As to the possibility of less size effect in quenched steels, we have recently completed a series of tests on S.A.E. 4340 steel in sizes ranging from $\frac{1}{8}$ to $1\frac{3}{4}$ in. in diameter for both notched and unnotched specimens. This steel heat treated to a tensile strength of 164,000 psi. exhibited a rather pronounced decrease in fatigue strength as the size of specimen was increased. The notch-sensitivities, as indicated by strength reduction factors for geometrically similar notched specimens, did not vary appreciably for the various sizes tested. Thus, the results of these tests did not confirm the predictions suggested by Mr. Peterson from statistical analyses. There did not appear to be any pronounced difference in the amount of scatter in fatigue life obtained from small as compared with large specimens; moreover, in the present tests there were no consistent differences in the amount of scatter from rapidly quenched steels as compared with the same steels slowly quenched. It appears probable that the statistical approach might be more useful if the concepts could be modified to take into account the alterations in properties of the metal which occur throughout the duration of a fatigue test.

Mr. Lessells and Mr. Jominy raise questions regarding the amount of residual stresses produced by the quench and remaining in the surfaces of the fatigue specimens. Because of time limitations in the oral presentation, it was not mentioned that the test specimens were roughly machined to $\frac{1}{2}$-in. diameter bars in the majority of cases and to $\frac{5}{8}$ in. diameter in the remaining specimens prior to heat treatment. They were then quenched and drawn and finally machined to the small size test bars. Most of the tempering temperatures were rather high, upwards of 1000 F. for all except one group of air-quenched samples. Thus we feel that the majority of the residual stresses, at least those of the macro type, should have been minimized by the stress relief occurring during tempering and by the removal of surface layers from these small diameter bars after the heat treatment. In answer to the specific question raised by Mr. Lessells, none of the tempered steels showed an abnormal amount of scatter in the fatigue tests, though no one knows what effect microresidual stresses may have on the results of fatigue tests.

The authors heartily agree with Mr. Knowlton and Mr. Kosting in the viewpoint that we seldom get 100 per cent martensite in commercial practice, and it is probable that some of the rapidly quenched steels discussed in this paper were not quenched to 100 per cent martensite. There was fairly definite evidence that water quenching the $\frac{1}{2}$-in. diameter bars of S.A.E. 1045 steel did not produce 100 per cent martensite. Therefore, in the paper we have tried to refer to the steels in many cases as "rapidly quenched," or to speak of them as primarily tempered martensite. As Mr. Jominy points out, rapid "quenching" is a relative phrase that needs to be interpreted in terms of the hardenability of the steel; hence, the term is not adequately descriptive to be sufficiently accurate. Perhaps it would be preferable to call it a "martensitic type" of structure. As far as the fatigue properties are concerned, the authors feel that the structures containing very finely dispersed carbides (martensitic type of structure) exhibited superior fatigue properties even though *not* representative of a structure that was fully martensitic on quenching. As Mr. Kosting has pointed out, those steels having relatively high transition temperatures (and inferior qualities) in the Charpy test probably were not 100 per cent martensitic, but apparently the fatigue properties of these steels with finely dispersed carbides were practically as good as those obtained for the metals with structures representative of 100 per cent martensite before tempering. To emphasize Mr. Kosting's comment with regard to the finite fatigue life of notched specimens, a comparison of Figs. 12 and 16 indicates that there appeared to be no correlation between the toughness and brittleness as indicated by a Charpy test and the finite fatigue life of notched specimens. The rapidly quenched groups of steels had similar properties, both as regards finite fatigue life and fatigue strength; however, there were undoubtedly constituents present other than martensite which did lead to wide alterations of Charpy impact properties for these same steels.

Practically all machine parts in service contain stress raisers in the form of keyways, holes, screw threads, fillets, etc.; thus the *notched* specimens which indicated an increase of 11 to 44 per cent in fatigue strength of the rapidly quenched steels as compared with the same metals slowly quenched were probably more representative of service conditions than the data from polished specimens. This might be interpreted as an indication of much greater notch-sensitivity in steels whose metallurgical structures were made up of coarser constituents such as pearlite plus ferrite, or conversely an improvement in the fatigue properties for structures that contained very finely dispersed carbides, regardless of whether the structures were 100 per cent tempered martensite. With such pronounced differences in fatigue strength it is not always economically feasible to improve the strength of a ("slowly quenched") part by increasing its diameter, particularly in those cases where weight is of great importance such as in the aircraft industry. It would seem desirable, therefore, to concentrate on heat treatments giving the optimum fatigue properties, if this could be accomplished without developing undesirable types of residual stresses.

Mr. Riegel has raised some pertinent questions regarding the applicability of laboratory tests to predict performance of a machine component in a given service condition. Undoubtedly, our present methods of laboratory experimentation and interpretation are inadequate in many instances to predict the behavior of a given material in a member in service. This is true for two reasons: (1) the inadequate knowledge of the actual constraints and conditions imposed in service; and (2) the lack of ingenuity in devising test procedures to study the behavior of materials. It is still desirable to test full scale models or components under actual service conditions, but for large structures this becomes impractical or impossible before the finished product is put in service. However, as far as the most highly stressed portions of the material are concerned these minute volumes of metal do not know whether they are in the laboratory or whether they are in a critical portion of a machine component in service. Hence, if we could produce in the laboratory all the environmental conditions of temperature, atmospheric medium, stress state, velocity of straining, range of stress, etc., that are normally encountered in service, it would be possible to observe the reactions of the material to this situation and to discriminate between those materials which offer the greatest resistance to the conditions encountered in normal service. The investigators, technologists, and engineers interpreting the results of an investigation must be continually on the alert to detect and appraise differences in the imposed laboratory conditions from those encountered in service. As Mr. Riegel has stated, "the lack of similarity of the modes of stressing probably accounts for the inability" to correlate Charpy impact with fatigue tests and also accounts for our inability to evaluate the suitability of certain metals for applications in specific machine parts. We often base our selection of these materials on static tension, Charpy impact, and rotating-beam fatigue tests; none of these tests are truly representative of the actual constraint and conditions to be encountered by the part in service. Only future developments in testing methods can rationalize our criteria for the selection of materials for structural components.

FATIGUE AND STATIC LOAD TESTS OF AN AUSTENITIC CAST IRON AT ELEVATED TEMPERATURES*

By W. Leighton Collins[1]

Synopsis

Repeated-load and short-time static load tests of unnotched and notched specimens machined from large plates of one heat of a commercial austenitic cast iron designed for high-temperature use are reported. Very little difference between the static tensile strengths of unnotched and notched (circumferential V-groove) specimens was observed at any temperature from room temperature to 1100 F. The endurance limits for unnotched and notched (small transverse hole) specimens for completely reversed cycles of bending stress were determined for five temperatures ranging from room temperature to 1160 F. The endurance limits for the two types of specimens varied only slightly at any test temperature, provided that the net cross-section is used in computing the stress in the specimens with the notch (hole). The results of the tests are compared to the results published in 1941[2] for similar tests of a high-strength process cast iron.

The static and fatigue properties of an austenitic cast iron at elevated temperatures were determined because of the interest in such tests expressed when similar data for a high-strength process cast iron were published in 1941.[2] An austenitic cast iron, one specifically designed for high-temperature use, was procured and some specimens were machined, but actual testing was interrupted by the war. The tests of notched and unnotched or notch-free specimens subjected to static and to repeated loads have now been completed and are reported in this paper. All of the tests are similar to those made on the process iron and consequently the new data, for the austenitic iron, are compared to the data published[2] for the process iron.

TABLE I.—CHEMICAL COMPOSITION.

Element, per cent	Austenitic Iron	Process Iron[a]
Total carbon	2.63	2.84
Combined carbon	0.67	0.74
Graphitic carbon	1.96	2.10
Silicon	2.14	1.52
Manganese	1.23	1.05
Copper	6.94	0.37
Chromium	2.09	0.31
Nickel	14.90	0.20
Molybdenum	0.00	0.00
Titanium	0.022	0.020
Vanadium	0.005	0.005
Arsenic	0.012	0.013
Phosphorus	0.16	0.07
Sulfur, evolved as H_2S	0.059	0.106
Sulfur, gravimetric	0.065	0.124

[a] See footnote 2.

Material and Test Specimens

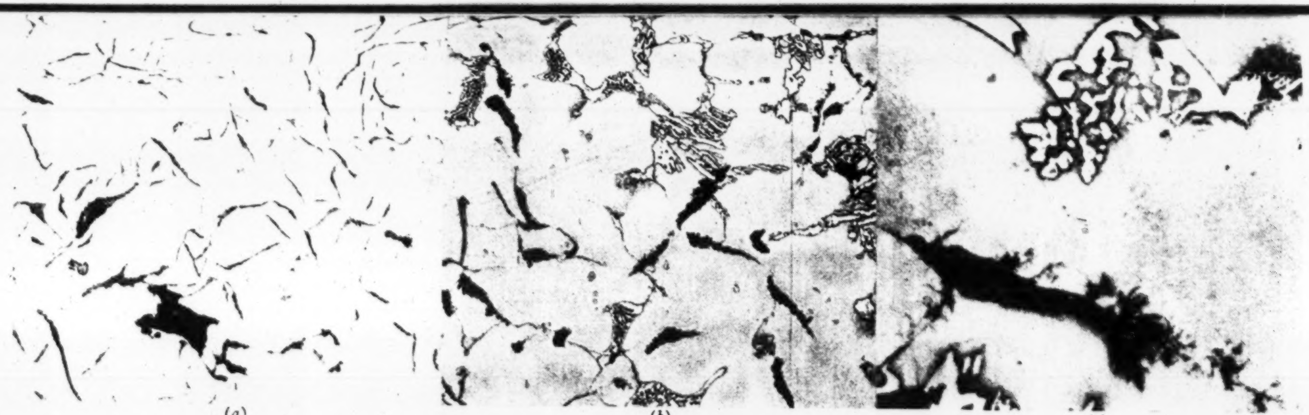
The material used was in the form of vertically cast plates, 1¾ in. thick, 26 in. long, and 16 in. high. All plates came from one pouring of iron and were poured

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

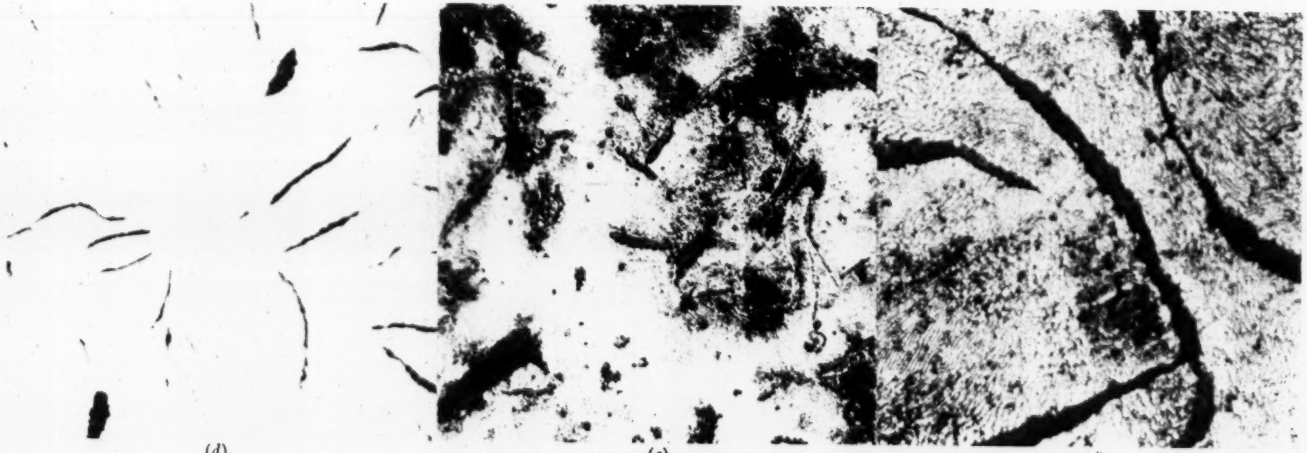
[1] Associate Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] W. Leighton Collins and James O. Smith, "Fatigue and Static Load Tests of a High-Strength Cast Iron at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 797 (1941).



(a) (b) (c)

Austentic Cast Iron



(d) (e) (f)

Process Cast Iron

FIG. 1.—Typical Photomicrographs.

Specimens taken from one-quarter point of thickness near center of casting.

(a) and (d)..................Unetched (× 100) (b) and (e)..................Etched in 2 per cent nital (× 100) (c) and (f)..................Etched in 2 per cent nital (× 500) from the top. The iron was always used in the "as cast" condition. The chemical composition of the iron, which can be classified as a high-nickel-copper-chromium iron of the austenitic type, is given in Table I. For the purpose of comparison, the chemical composition of the slightly alloyed nickel-chromium-copper process iron used in the previous tests[2] is also given.

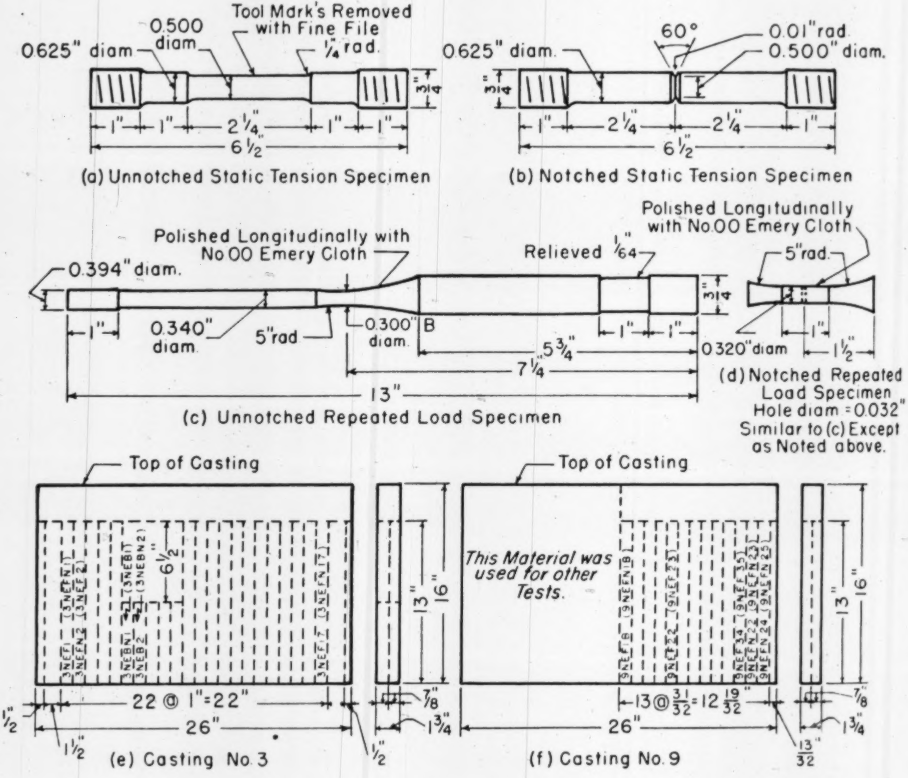

0625" diam
0.500 diam
Tool Mark's Removed with Fine File
¼" rad.
3/4"
1" 1" 2¼" 1" 1"
6½"
(a) Unnotched Static Tension Specimen
0.625" diam.
60°
0.01" rad.
0.500" diam.
3/4"
1" 2¼" 2¼" 1"
6½"
(b) Notched Static Tension Specimen
0.394" diam.
Polished Longitudinally with No.00 Emery Cloth
Relieved 1/64"
0.340" diam.
5" rad
0.300" diam.
B
1" 1" 1"
5¾"
7¼"
13"
(c) Unnotched Repeated Load Specimen
Polished Longitudinally with No.00 Emery Cloth
5" rad.
0.320" diam
1"
1½"
(d) Notched Repeated Load Specimen
Hole diam = 0.032"
Similar to (c) Except as Noted above.
Top of Casting
6½"
13"
16"
22 @ 1" = 22"
26"
½"
1½"
⅞"
1¾"
(e) Casting No. 3
Top of Casting
This Material was used for other Tests.
13 @ 31/32 = 12 19/32"
26"
13"
16"
⅞"
1¾"
13/32"
(f) Casting No. 9

FIG. 2.—Specimens and Castings.

Parentheses around specimen number means the specimen came from the back half of the thickness of the casting.

The typical microstructures of the two irons are shown in Fig. 1. The specimens for the photomicrographs came from the same location in the thickness of a casting as did the centers of the test specimens and thus are representative of the material at the center of the test specimens. In accordance with the A.S.T.M.-A.F.A. Charts for Evaluating the Microstructure of Graphite in Gray Iron,[3] both irons were of type *A*, random orientation of graphite flakes, and the graphite flakes were, in general, size 4, 0.5 to 1.0 in. in length, although the austenitic iron did have some flakes over 1 in. in length, size 5. Also, the austenitic iron was a class 30[4] iron, whereas the process iron was of class 40.[4] The photomicrographs of both irons, when etched in 2 per cent Nital and magnified 500 diameters, see Fig. 1, indicate that the high-strength iron was a relatively fine pearlitic iron with no free ferrite and that the austenitic iron had the graphite at the austenite grain boundaries. The irregularly shaped areas in the austenitic iron apparently are of eutectic composition.

---

[3] Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron (A 247 - 47), 1946 Book of A.S.T.M. Standards, Part I-A, p. 1073.

[4] Standard Specifications for Gray-Iron Castings (A 48 - 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 593.

The four types of specimens used and their dimensions are shown in Fig. 2. They are identical to those used in the previous tests except for the diameters of the small ends of specimens (*c*) and (*d*) which were reduced from 0.500 in. to 0.340 in. for most specimens so as to increase their deflection when loaded and thus facilitate automatic stopping of the machine when fracture occurred. Specimen (*a*) is in accordance with the A.S.T.M. Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 - 43)[5] and was used for the unnotched short-time static tension tests at all temperatures. Specimen (*b*) is the notched (circumferential 60-deg. V-groove having a depth of 0.0625 in. and a radius at the root of the notch of 0.01 in.) specimen tested similarly to (*a*) in order to determine notch sensitivity under short-time static tensile loads at various temperatures. Specimens (*c*) (unnotched) and (*d*) (notched with a transverse, diametral hole having a diameter of one-tenth the diameter of the specimen) were used for the repeated-load tests at elevated temperatures.

The location of each specimen in the casting also is shown in Fig. 2. The first number for each specimen designates the number of the casting from which the specimen came; the other numbers and letters indicate type of specimen, kind of test, and location in the casting. The repeated-load specimens came from two castings, but since such an arrangement apparently had no significant effect in previous tests it is not believed to be significant here.

EQUIPMENT AND TEST PROCEDURE

Static Tests:

The static load tests were made in a 10,000-lb. motor-driven Olsen screw-power testing machine at a free speed of loading head of 0.075 in. per min. The apparatus used and the test procedures were the same as described in the report on the process iron[2] and consequently are not discussed here. In all cases the specimens were placed in an electric furnace mounted on the testing machine and brought to temperature in 2 to 2½ hr. by a current not exceeding by more than 25 per cent that required to maintain the test temperature, except for the notched specimen at 400 F. for which the maximum current exceeded the average to maintain temperature by 50 per cent. The test temperature was maintained within ±2 to ±6 F. for 30 to 45 min. prior to starting the application of load in order to make certain of approximate thermal equilibrium, except for the notched specimen at 402 F. for which the time was 1 hr. 30 min. The temperature gradient over the reduced length of 2½ in. was determined by 3 thermocouples and was 10 to 15 F., or less, for the 30 or more minutes at constant temperature. The test temperature reported is the temperature at the center of the reduced length or, for the notched specimens, at the root of the notch.

Fatigue Tests:

The repeated-load tests at all temperatures were made in a constant strain type of fatigue testing machine[6] subjecting the specimen to completely reversed cycles of bending stress and operating at a speed of 2500 rpm. The machine used and the testing procedure were the same as for the previous tests[2] except that whenever a specimen was inserted in the machine or the deflection was being checked, a dead load was used to stress the specimen instead of a calibrated spring. After the specimen was loaded, the deflection was kept constant by clamping the bearing holding the small end of the specimen to the rotating part of the machine; the other end of the specimen was held firmly in a vise. At frequent intervals during the test the machine was stopped, the clamp was loosened and the deflection produced by the desired load was checked. In this manner the load was kept approximately constant throughout the entire test.

---

[5] 1946 Book of A.S.T.M. Standards, Part I-A, p. 728

[6] Fully described by H. F. Moore and G. N. Krouse, "Repeated Stress (Fatigue) Testing Machines Used in the Materials Testing Laboratory of the University of Illinois," *Circular No. 23*, Engineering Experiment Station, University of Illinois (1934); also, H. F. Moore and N. J. Alleman, "Progress Report on Fatigue Tests of Low-Carbon Steel at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 114 (1931).

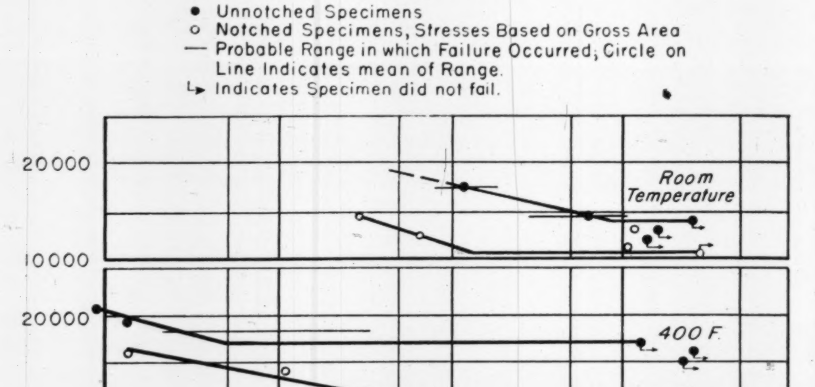


FIG. 3.—S-N Diagrams for Completely Reversed Cycles of Bending Stress.

In all tests the specimen was brought nearly to the test temperature by a current slightly in excess of that required to maintain the desired temperature. The current was then reduced so that the specimen reached the test temperature by the time loading was completed. The application of the dead load, the clamping of the bearing, and the starting of the machine were always completed as rapidly as possible in order to prevent creep from causing any of the measured deflection or producing a permanent curvature of the specimen. The variation in stress during a test never exceeded 500 psi. The variation in temperature from the mean over a 1-in. length containing the critical section of the specimen normally did not exceed ±5 F. and in no case exceeded ±10 F., except for very short periods of time when the machine was started or stopped. The temperature of the furnace was maintained within a range of ±7 F. Because of the peculiar behavior of the specimens and the difficulties with the cut-off switch, some specimens had to be reheated and the machine started again, but for only two specimens did cooling and reheating occur more than once.

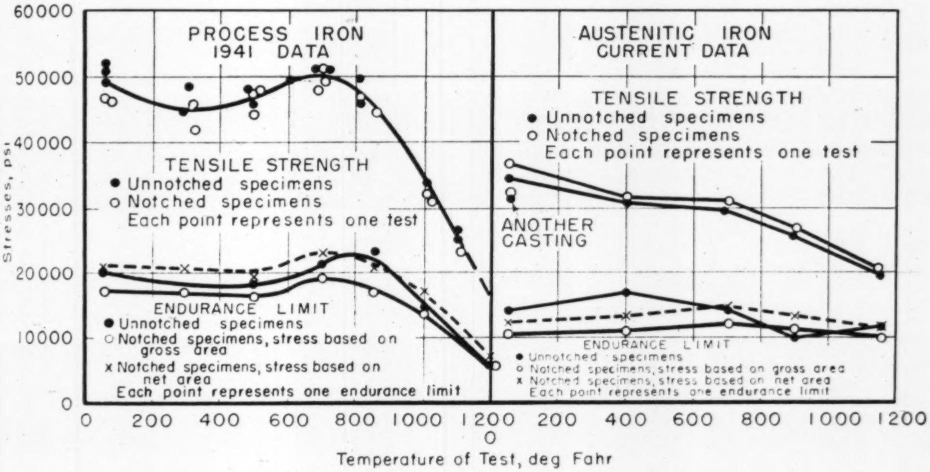


FIG. 4.—Strength-Temperature Curves.

TEST DATA AND RESULTS

Static Tests:

The tensile strengths obtained from the short-time tests of unnotched and notched specimens are shown in the right-hand portion of Fig. 4. The stress for the notched specimen is based on the area at the root of the circumferential V-groove. For each of the five test temperatures one specimen of each type was tested. The points for each type are connected by straight lines because the data are not sufficient to establish an exact law of behavior. Additional tests could not be made because there was no more material.

The notched specimens had tensile strengths from 3 to 7 per cent greater than the unnotched specimens for all temperatures from room temperature to 1160 F. This consistent difference is small and undoubtedly is less than the spread in results that would have been obtained if several specimens of any one type had been tested at a given temperature. Although there may be some doubt about the correctness of assuming a normal distribution curve, a statistical analysis was made using the method outlined by Fisher.[7] The probability of the difference between the static strengths of unnotched and notched specimens occurring by chance is less than 0.01. Thus the differences are considered to be significant even though they are small. Obtaining the higher static strengths for the notched specimens of this austenitic iron is just the opposite of the results obtained for the previously tested process iron.[2] The difference most probably is due to the fact that the austenitic iron has a much greater ability to deform (has more ductility) than the process iron, as determined from static load tests at room temperature.[8] The results of the tests of the process iron also are shown in Fig. 4 in order that the data for the two irons can be compared. It will be noted that at about 1160 F. the austenitic iron had a tensile strength of about 55 per cent of its strength at room temperature, whereas the process iron had about 40 per cent of its room temperature strength at about 1160 F.

---

[7] R. A. Fisher, "Statistical Methods for Research Workers," Sixth Edition, p. 186, Oliver and Boyd, London (1936).

TABLE II.—ENDURANCE LIMITS.

Temperature, deg. Fahr.	Endurance Limits, psi.		
	Unnotched	Notched[a]	
		Gross Area	Net Area
Room	14 000	10 500	12 600
400	17 000	11 000	13 200
700	14 000	12 200	14 600
900	10 000	11 000	13 200
1160	12 000	10 000	12 000

[a] Notch consists of a transverse hole whose diameter is 0.10 the diameter of the specimen.

Fatigue Tests:

The *S-N* curves for the repeated-load tests at the five temperatures used are shown in Fig. 3. The stresses for the notched specimens are based on the gross area, no allowance having been made for the material removed in forming the hole. All of the endurance limits are tabulated in Table II. The values for the notched specimens tabulated under the heading "net area" are computed on the basis of having the holes all the way through the specimens even though many of the holes had a depth of about half the radius. The maximum error in the computed stress resulting from that assumption does not exceed one per cent. In most instances the holes could not be drilled all the way through the specimens without having the small drills break off in the hole. Consequently holes usually were drilled from each side of the specimen and drilling was stopped if a good hole having a depth of about half the radius was obtained. Specimens with the deepest holes were almost always used for the stresses at which fracture was not expected.

All endurance limits except one, see Fig. 3, are based on at least 20,000,000 cycles of stress, and for all *S-N* curves the difference between the stress in the lowest stressed specimen that broke and the endurance limit did not exceed 1000 psi. It will be observed that the endurance limit for the notched (0.032-in. diam. hole) specimens are considerably less than for the unnotched specimens at all temperatures except 900 F., where the reverse is true. The author has no explanation for this variation from the normal behavior or for the unusually large difference in endurance limits found at 400 F. Just as in other tests,[2, 7] the *S-N* curves are very flat, and specimens tested at stresses of 3000 to 4000 psi. above the endurance limits broke at relatively small numbers of cycles; this means that any overloading, even though it is for a relatively small number of cycles, may lead to an early failure.

The endurance limits plotted against temperature are shown in the lower right-hand part of Fig. 4. It should be noted that the endurance limits for the notched specimens are plotted using both the gross and the net cross-sections in computing stresses. Although specimens came from two different castings (see Fig. 2), it is not believed there was sufficient difference in their strengths to influence the results.

---

[8] W. Leighton Collins and James O. Smith, "The Notch Sensitivity of Alloyed Cast Irons Subjected to Repeated and Static Loads," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 639 (1942).

The endurance limits are connected by straight lines in Fig. 4 because it is not believed their variation establishes an exact law of behavior. This is particularly true for the unnotched specimens because they exhibit somewhat of an unordered variation with temperature. If the endurance limits for the unnotched and notched specimens are both considered, the stress in the notched specimen being based on the net area, it is apparent that the general shape of the endurance limit – temperature curve is not the same as the static tensile strength—temperature curve; the endurance limit decreases only slightly as temperature increases, particularly for the notched specimens, whereas the static tensile strength decreased considerably, particularly above about 700 F. Since the data for endurance limits based on net areas coincide reasonably well with the data for the unnotched specimens, it is evident that the stress concentration factor is almost unity for all temperatures used.

TABLE III.—COMPARISON OF ENDURANCE LIMITS AND TENSILE STRENGTHS.

Temperature, deg. Fahr.	Average Endurance Limit, psi.[a]	Average Tensile Strength, psi.[b]	Endurance Ratio
Room	13 300	35 500	0.37
400	15 100	31 400	0.48
700	14 300	30 400	0.47
900	11 600	26 400	0.44
1160	12 000	19 900	0.60

$$\text{Endurance ratio} = \frac{\text{Average endurance limit at a given temperature}}{\text{Average tensile strength at the same temperature}}$$

[a] Average of endurance limits of unnotched specimens and of notched specimens, based on net cross-section.

[b] Average of unnotched and notched specimens.

If the differences in endurance limits for unnotched and notched (stress based on net area) specimens are studied by the method outlined by Fisher[7] (again realizing there may be some doubt about the correctness of assuming a normal distribution curve), the probability of the differences occurring by chance is between 0.80 and 0.90. Thus the differences are not considered to be significant. The conclusion that the stress concentration factor is approximately unity for all temperatures used, provided the stress in the notched specimen is based on the net area, is the same as that reached for the process iron.[2] However, for the process iron[2] the endurance limits for notched specimens based on net areas were usually slightly greater than the endurance limits for unnotched specimens, whereas no such marked trend was found for the austenitic iron. It is possible that this difference may be due to the greater ability to deform of the austenitic iron.[8]

The difference in the magnitude of the effect of a high temperature on the endurance limits of an austenitic and a process iron is shown by the fact that at 1160 F. the average endurance limit (average of the endurance limits of unnotched and notched, based on net cross-sections, specimens) of the austenitic iron is about 90 per cent of the room temperature average endurance limit whereas the average endurance limit of the process iron at the same temperature is about 40 per cent of the room temperature average value.

The endurance ratio of a material is usually defined as the ratio of the endurance limit to the tensile strength, both values being for the same temperature. Such a ratio was computed for each of the five test temperatures used and the values are shown in Table III. In the computations the average of the endurance limits for unnotched and notched specimens was used, the stress for the notched specimen being based on the net area.

If the unusually low value of 0.44 for 900 F. is neglected, the endurance ratio increases approximately linearly from 0.37 at room temperature to 0.60 at 1160 F. The maximum differences from such a variation is +9 per cent to −2 per cent, except at 900 F. where the difference is −19 per cent. The large difference at 900 F. is believed to be due to the unusually low endurance limit found for unnotched specimens at that temperature and indicates that additional tests should be made before the results can be considered entirely conclusive. The variable value of endurance ratio for the austenitic iron is different from the almost constant value of 0.44, with a maximum difference of ±9 per cent, found for the process iron.[2]

The length of time the specimen was in the furnace had little visible effect on the specimen. Although there was some discoloration there was no scaling. In all other respects the failures were typical of what would be expected of a brittle material subjected to repeated loads. All the notched specimens that failed fractured at a section through the notch; the unnotched specimens that failed fractured at sections so close to the critical section in almost all cases that no adjustment of stress values seemed necessary or desirable.

CONCLUSIONS

From a study of the data obtained for the austenitic cast iron and presented in the tables and figures and from a comparison of those data with the data obtained for a similarly tested high-strength process cast iron,[2] the following conclusions can be reached regarding the effect of elevated temperatures on the fatigue and static tensile strengths of an austenitic cast iron.

Short-Time Static Tension Tests:

1. A circumferential V-groove did not decrease the static tensile strength of the austenitic cast iron at any temperature ranging from room temperature to 1160 F. In fact, the tensile strengths of the notched specimens were about 5 per cent higher than the values obtained for the unnotched circumferential V-groove, specimens.

2. The average static tensile strength of unnotched and notched specimens of the austenitic cast iron varied from 35,500 psi. at room temperature to 30,400 psi. at 700 F. and then to 19,900 psi. at 1160 F.

Fatigue Tests in Bending:

3. For completely reversed cycles of bending stress, a transverse hole having a diameter of 0.10 the diameter of the specimen had very little effect on the fatigue strength of the austenitic cast iron, at temperatures ranging from room temperature to 1160 F., provided the stresses in the specimens containing the holes are calculated on the bases of the net cross-sections at the holes.

4. For the austenitic cast iron the endurance limit for completely reversed cycles of bending stress as determined from unnotched and notched specimens, based on net cross-section, did not vary greatly for temperatures ranging from room temperature to 1160 F.; from room temperature to 700 F. the average endurance limit was about 14,000 psi. and at 1160 F. it was 12,000 psi.

5. For the austenitic cast iron the endurance ratio, defined as the ratio of the endurance limit for completely reversed cycles of bending stress to the static tensile strength at the same temperature, increased from 0.37 at room temperature to 0.60 at 1160 F., except for an unusually low value of 0.44 at 900 F.

Comparison of an austenitic and a process cast iron:

6. The austenitic and the process cast irons showed little effect of a circumferential V-groove on the static tensile strength at any temperature ranging from room temperature to 1160 F.; for the austenitic iron the values for the notched specimens were a little higher than for the unnotched specimens, whereas the opposite was true for the process iron.

7. The static tensile strength of the austenitic iron at 1160 F. is about 55 per cent of the room temperature static tensile strength, whereas the similar value for the process iron was about 40 per cent.

8. The average endurance limit for unnotched and notched specimens, based on net area, of the austenitic iron at 1160 F. was about 90 per cent of the room temperature average endurance limit, whereas the similar value for the process iron is about 40 per cent.

9. The austenitic and the process irons both showed little effect of a small diametral hole on the endurance limit at any temperature ranging from room temperature to 1160 F., provided the stresses in the specimens containing the holes are calculated on the basis of the net cross-sections at the holes.

10. For the austenitic cast iron the endurance ratio, defined as the ratio of the endurance limit for completely reversed cycles of bending stress to the static tensile strength at the same temperature, increased from 0.37 to 0.60 as the temperature increased from room temperature to 1160 F., whereas it remained at an almost constant value of 0.44 for the process iron.

11. Maximum values of static tensile strength and endurance limit of the austenitic cast iron may be said to have been obtained at room temperature, whereas the maximum values for the process iron were obtained at 700 to 800 F.

Acknowledgments:

The work herein reported was carried out as a project of the Engineering Experiment Station in the Department of Theoretical and Applied Mechanics of the University of Illinois and was partially financed by funds obtained from the A.S.T.M. through Subcommittee XXII on Elevated Temperature Properties of Cast Iron, of Committee A-3 on Cast Iron.

Most of the laboratory work was done by H. T. Corten, Research Graduate Assistant, and C. E. Spaeth, Senior Student in Mechanical Engineering. Acknowledgment is made to The International Nickel Co., Inc. and to J. S. Vanick of that company for their interest in the project and for providing the iron, and to J. T. MacKenzie of the American Cast Iron Pipe Co. for his cooperation in providing the chemical analyses and photomicrographs.

DISCUSSION

Mr. E. A. Imbembo.[1]—Mr. Collins has indicated that for the repeated stress specimen a drilled hole was used as the stress raiser and that for the tension specimen a circumferential V-notch was used. What factors led to the selection of the type of stress raiser used in the repeated stress specimen, and why were different notches employed for the two tests?

Mr. P. R. Toolin.[2]—I should like to have Mr. Collins' opinion as to whether or not the cast irons have a definite fatigue limit at the higher test temperatures.

Mr. George R. Gohn.[3]—I wonder whether it might not be advisable to speak of the various values as fatigue strengths at a definite cycle life rather than endurance limits? This is in line with the efforts of A.S.T.M. Committee E-9 on Fatigue to standardize on the use of these terms.

I understand that while the ratio between fatigue strength and tensile strength was higher for the austenitic cast iron, the absolute fatigue values for the austenitic and for the process iron were about the same at 1160 F. At lower temperatures the fatigue strengths of the process iron were somewhat higher than the corresponding values for the austenitic iron. Is that correct?

Mr. Alan H. Harris.[4]—I should like to ask Mr. Collins whether these cast iron samples were heat treated, and if not, whether he would hazard a guess as to the effect of the heat treatment on the fatigue life of process iron.

Mr. R. W. Mason.[5]—There has been some work done recently by T. E. Eagan[6] of Cooper-Bessemer which indicates that heat treatment of cast irons does not greatly affect the endurance limit even though the tensile strength may be much increased.

I should like to ask whether there was any appreciable amount of oxidation on any of the samples in the time they were held at the elevated temperatures? If there was, is it the author's opinion that that amount of oxidation affected the results in any way?

Mr. J. W. Bolton.[7]—In respect to Mr. Collins' test, the effect of time in relationship to effects of temperature is left out. Regular cast irons deteriorate when exposed for long periods at temperatures above 750 F., for example, at 900 F. Austenitic cast irons do not deteriorate. Therefore Mr. Collins' results are invalid for other than the relatively short periods of test he employed. The author's closure should emphasize that the data and conclusions are not applicable under usual conditions of engineering service.

Mr. W. Leighton Collins (*author's closure*).—Mr. Harris's question about heat treatment is answered by saying that neither cast iron was heat treated; all specimens were machined directly from the castings in the "as cast" condition and then tested. In the original plan for the study of the process iron the effect of heat treatment was included, but since machining was impossible for the treated iron that was received that part of the investigation had to be dropped. Mr. Mason's discussion provides a source of information regarding the effect of heat treatment on the fatigue life of process iron and the author thanks him for providing a direct reference.

---

[1] Senior Metallurgist, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

[2] Mechanics Dept., Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.

[3] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[4] Engineering Division, Chrysler Corp., Detroit, Mich.

[5] Metallurgist, The International Nickel Co., Inc., Detroit, Mich.

[6] T. E. Eagan, "Effects of Heat Treatment on the Endurance Limit of Alloyed Gray Cast Iron", *Transactions*, Am. Foundrymen's Assn., Vol. 54, p. 230 (1946).

[7] Director of Metallurgical Research and Testing, The Lunkenheimer Co., Cincinnati, Ohio.

Mr. Imbembo asked why different types of notches were used in the static and repeated load tests. When the elevated temperature tests were being planned an investigation of the notch sensitivity of cast irons was under way[8] and the types of notches selected were made to agree with those already being used. The diametral hole was used because it was most desirable for tests in torsion; the V-groove for the axial load tests was selected in order to more nearly obtain axiality of loading.

Mr. Bolton is entirely correct in saying that the effect of time in relationship to effects of temperature is not considered in the paper. The omission of such an important phase of elevated temperature studies was due only to the limitation on the amount of work that could be included in the project. It was not intended to infer that that time was unimportant, or that the data and conclusions would be applicable for the long periods of time usually encountered under service conditions, or that a direct extrapolation to such conditions could be made. It is highly desirable to extend the investigation to include the effect of time, both by increasing the number of cycles of stress at the rate of application used in the current tests and by reducing the number of applications of stress per unit of time, for without such an extension little can be done to study the effect of instability of the material or of the relative significance of repeated loading and creep in causing fracture.

A program involving the effect of time is also required to answer the points raised by Mr. Toolin and Mr. Gohn regarding the existence of a definite endurance limit. At elevated temperatures, and particularly if a long period of time is involved, the behavior of a material is a combination of creep and repeated loading. Because the total time of test was kept short in the tests, it was believed that repeated loading would be the most significant factor leading to fracture. Consequently the *S-N* curves were drawn in the conventional manner, showing a "kink," and the term "endurance limit" was used. Since it is probable, particularly for the process iron, that fracture might occur at stresses below those reported as "endurance limits" if a longer period of time was used than that required for 20,000,000 cycles, Mr. Gohn's suggestion to use "fatigue strength at 20,000,000 cycles" may be a good one.

The data for answering Mr. Gohn's question regarding the variation in ratio of endurance limit to tensile strength and the variation in the absolute endurance limits is given in Fig. 4 and Table III. Mr. Gohn is correct in his general interpretation: the endurance limits for the process iron were greater than similar values for the austenitic iron for all temperatures up to about 1100 F.; the static strength of the process iron was greater than the static strength of the austenitic iron for temperatures up to about 1160 F., the maximum value used in the tests, at which temperature the two materials would seem to be of about equal strength.

---

[8] W. Leighton Collins and James O. Smith, "The Notch Sensitivity of Alloyed Cast Irons Subjected to Repeated and Static Loads," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 639 (1942).

Mr. Mason asked about oxidation. To the unaided eye the specimens of the austenitic iron showed no change in surface appearance for any temperature used, but the specimens of the process iron discolored considerably, particularly for the higher temperatures. Since the structure of the irons after exposure to temperature was not studied, a definite answer to the effect of oxidation on the results cannot be given; it would, however, seem reasonable to believe that oxidation might have had some influence on the results of the tests, particularly for the process iron.

FATIGUE PROPERTIES OF SOME COPPERS AND COPPER ALLOYS IN STRIP FORM*

By H. L. Burghoff[1] and A. I. Blank[1]

Synopsis

The results of reversed-bending fatigue tests on three types of copper, five copper-zinc alloys, and four other copper-base alloys in the form of 0.032-in. strip are given in this paper. The results are correlated in graphs showing the relationships between fatigue strength, tensile strength, composition, degree of reduction by cold rolling, grain size, and angle of applied stress with respect to the rolling direction.

Additions of phosphorus up to at least 0.027 per cent increase the fatigue strength of copper, and reduction of 37 per cent by rolling gives higher fatigue properties than reductions of 21 and 60 per cent.

Fatigue strength of the alpha brasses increases with zinc content up to 15 per cent zinc and does not change appreciably with increasing zinc content except for a minor decrease at 35 per cent zinc. Fatigue strength of some of the brasses increases with degree of reduction from the annealed condition to 60 per cent reduction. Fatigue strength of the other brasses increases initially with cold working but does not change significantly beyond moderate reductions. There is a limited proportional relationship between fatigue strength and tensile strength. Fatigue strength of hard-rolled brasses increases as the angle with the rolling direction increases.

Fatigue strength of the annealed materials decreases as the grain size increases. Similarly, fatigue strength of the hard-rolled materials decreases as the ready-to-finish grain size increases, although the effect diminishes with increasing degree of reduction.

Fatigue strength of copper is increased when it is alloyed with tin. Additions of tin to red brass and of lead to high brass have no significant effect on the fatigue properties of these alloys in strip form.

Copper alloys have been widely used in numerous applications requiring a combination of properties such as ease of forming, resistance to corrosion, electrical conductivity, and fatigue strength. Most applications of this kind, as in the communications field, employ flat springs made from sheet or strip metal of relatively thin gage.

The present work describes the fatigue properties of three types of copper, five copper-zinc alloys, and four other copper-base alloys. Correlations are included showing the effect of phosphorus content on the fatigue strength of copper and, similarly, the effect of zinc content in the brasses. The variations of fatigue strength with degree of reduction by rolling, with grain size of annealed materials, with ready-to-finish grain size, and

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Research **Metallurgist and** Assistant Research Metallurgist, **respectively,** Chase Brass and Copper Co., Waterbury, **Conn.**

TABLE I.—COMPOSITION AND TEMPER OF MATERIALS AND SUMMARY OF TEST RESULTS.

Material and Composition	Ready-to-Finish Grain Size, mm.	Tempers on 0.032-in. Gage: Rolled	Tempers on 0.032-in. Gage: Annealed	Angle with Rolling Direction, deg.	Rockwell Hardness		Stress, 0.2 per cent Offset, psi.	Stress at 0.5 per cent Elongation under Load, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Fatigue Strength at 1,000,000 Cycles, psi.	Fatigue Strength at 10,000,000 Cycles, psi.	Fatigue Strength at 100,000,000 Cycles, psi.	Ratio, Fatigue Strength at 100,000,000 Cycles to Tensile Strength
		Reduction, per cent	Grain Size, mm.		F	B								
Electrolytic Copper 99.93 Cu	0.030	21	....	0	81	34	....	39 000	41 000	17	19 000	13 500	12 000	0.29
	0.030	37	....	0	88	48	....	....	49 300	6	22 500	15 500	13 000	0.26
	0.030	60	....	0	93	58	....	....	57 700	4	23 000	14 500	11 000[a]	0.19
Deoxidized Copper Low Residual Phosphorus 99.97 Cu, 0.008 P	0.040	21	....	0	83	40	....	37 500	41 400	11	19 500	13 500	13 000	0.31
	0.045	37	....	0	87	50	....	41 900	45 300	4	21 500	16 000	15 500	0.34
	0.040	60	....	0	92	58	....	48 400	51 600	2	23 500	16 000	14 000	0.27
Deoxidized Copper High Residual Phosphorus 99.96 Cu, 0.027 P	0.030	21	....	0	82	37	....	40 300	42 800	17	20 000	14 000	14 000	0.33
	0.025	37	....	0	89	51	....	....	51 600	5	26 000	19 000	19 000	0.37
	0.030	60	....	0	94	61	....	....	59 300	2	24 500	13 500	18 500	0.31
Commercial Bronze, 90 per cent 89.74 Cu, 0.01 Fe, <0.005 Pb, 10.25 Zn (by diff.)	0.030	....	0.030	0	57	....	10 400	11 700	39 000	46	14 000	12 000[a]	10 000[a]	0.26[a]
	0.030	21	....	0	....	50.5	37 700	38 800	47 000	25	21 300	17 100	16 000	0.34
	0.030	37	....	0	....	68.5	53 500	53 000	59 700	7	25 500	17 700	16 500	0.28
	0.035	60	....	0	....	76.5	56 000	56 000	66 900	6	25 700	19 700	18 000	0.27
	0.035	68	....	0	....	80	65 000	60 000	73 300	5	29 000	20 500	20 000	0.27
Red Brass, 85 per cent 85.00 Cu, 0.015 Pb, 0.01 Fe, <0.005 Ni, 14.97 Zn (by diff.)	0.030	....	0.020	0	64	....	....	....	44 000	42	19 500	18 000	16 500	0.37
	0.030	....	0.075	0	51	....	....	....	39 000	44	15 000	12 700	10 500	0.27
	0.070	....	0.025	0	60	....	....	13 500	42 000	44	20 000	16 900	14 500	0.35
	0.070	....	0.090	0	48	....	....	8 800	38 500	44	16 500	12 500	11 000	0.29
	0.020	21	....	0		65	....	....	56 500	14	27 000	22 500	22 500	0.40
	0.020	21	....	45	....	65	....	48 400	52 000	13	29 500	23 000	23 000	0.44
	0.020	21	....	90	....	65	....	....	56 000	10	29 500	24 000	24 000	0.43
	0.030	37	....	0	....	78	....	59 700	69 000	7	32 500	22 500	21 000	0.30
	0.030	37	....	45	....	78	....	58 600	69 500	4.5	34 500	24 000	24 000	0.35
	0.030	37	....	90	....	78	....	64 500	75 000	5.5	35 500	29 000	29 000	0.39
	0.025	60	....	0	....	84	....	....	79 000	5	32 500	24 000	24 000	0.30
	0.025	60	....	45	....	84	....	63 400	80 500	3.5	38 000	27 000	27 000	0.34
	0.025	60	....	90	....	84	....	....	88 000	5	38 000	29 300	29 000	0.33
	0.060	21	....	0	....	58	....	45 800	50 000	17	22 500	15 000	15 000	0.30
	0.060	21	....	45	....	58	....	44 300	48 500	14	24 500	16 500	16 500	0.34
	0.060	21	....	90	....	58	....	45 600	50 000	12	24 000	18 000	18 000	0.36
	0.070	37	....	0	....	74	....	56 500	64 000	6	27 500	19 000	19 000	0.30
	0.070	37	....	45	....	74	....	55 600	63 000	6	27 000	18 000	18 000	0.29
	0.070	37	....	90	....	74	....	57 800	66 000	7	26 000	19 500	19 500	0.30
	0.070	60	....	0	....	84	....	....	79 000	4	30 500	23 500	23 500	0.30
	0.070	60	....	45	....	84	....	....	83 000	3.5	36 500	25 500	25 500	0.31
	0.070	60	....	90	....	84	....	....	91 000	4.5	38 500	28 500	28 500	0.31
Low Brass, 80 per cent 80.18 Cu, 0.01 Pb, 0.01 Fe, 0.005 Ni, 19.79 Zn (by diff.)	0.040	....	0.035	0	68.5		15 000	15 700	46 200	51	18 600	15 000	14 000	0.30
	0.040	21	....	0	....	74.5	55 000	55 400	63 600	14.5	25 500	19 000	19 000	0.30
	0.040	37	....	0	....	83	67 000	62 000	76 900	6.5	27 000	22 700	22 500	0.29
	0.035	60	....	0	....	89	75 000	62 500	88 000	5.0	29 200	22 000	22 000	0.25

---

[a] By extrapolation.

Cartridge Brass, 70 per cent 69.91 Cu, 0.015 Pb, 0.01 Fe, 0.005 Ni, 30.06 Zn (by diff.)	0.030	....	0.035	0	65	....	....	....	49 500	63	20 500	14 500	12 500	0.25
	0.030	....	0.100	0	52	....	....	....	45 000	68	18 000	12 300	10 500	0.23
	0.075	....	0.025	0	64	....	....	....	49 000	58	21 500	16 500	15 500	0.32
	0.075	....	0.120	0	49	....	....	....	45 000	64	18 000	12 000	11 500	0.26
	0.025	21	....	0	....	69	....	52 000	63 400	24	28 000	18 500	18 500	0.29
	0.025	21	....	45	....	69	....	51 700	61 300	22	31 500	20 200	20 000	0.33
	0.025	21	....	90	....	69	....	53 100	64 500	18	29 500	19 000	19 000	0.29
	0.035	37	....	0	....	82	....	....	76 500	8	33 500	22 000	22 000	0.29
	0.035	37	....	45	....	82	....	....	73 500	8	30 000	21 000	21 000	0.29
	0.035	37	....	90	....	82	....	62 800	80 500	8	31 500	24 000	24 000	0.30
	0.025	60	....	0	....	91	....	....	92 500	3.5	30 000	22 000	22 000	0.24
	0.025	60	....	45	....	91	....	64 200	97 000	3.5	36 000	24 500	24 500	0.25
	0.025	60	....	90	....	91	....	....	101 000	3.5	40 500	28 000	28 000	0.28
	0.080	21	....	0	....	70	....	....	62 000	32	23 500	18 000	18 000	0.29
	0.080	21	....	45	....	70	....	....	58 000	30	29 500	19 000	18 500	0.32
	0.080	21	....	90	....	70	....	....	61 000	....	29 000	19 000	19 000	0.31
	0.075	37	....	0	....	81	....	59 900	71 500	11	33 000	21 000	21 000	0.29
	0.075	37	....	45	....	81	....	58 200	71 000	8	27 500	20 000	20 000	0.28
	0.075	37	....	90	....	81	....	....	75 500	10	32 000	19 200	19 000	0.25
	0.075	60	....	0	....	90	....	66 400	92 000	3.5	30 500	24 000	24 000	0.26
	0.075	60	....	45	....	90	....	66 400	94 500	4	34 000	25 000	25 000	0.26
	0.075	60	....	90	....	90	....	69 900	99 000	3.5	42 000	28 300	28 000	0.28
High Brass, 65 per cent 65.22 Cu, 0.08 Pb, 0.02 Ni, 0.01 Fe, 34.67 Zn (by diff.)	0.030	....	0.030	0	75	....	19 600	20 000	51 700	50	23 500	16 500	15 000	0.29
	0.030	21	....	0	....	74	52 500	52 500	64 800	25.5	27 400	19 700	19 000	0.29
	0.030	37	....	0	....	85	73 300	63 000	80 700	6	28 300	19 000	19 000	0.24
	0.040	60	....	0	....	90.5	82 500	68 000	94 000	1.5	28 800	19 000	19 000	0.20
High-Leaded Brass 64.09 Cu, 2.03 Pb, 0.02 Fe, 0.02 Ni, 0.01 Sn, 33.83 Zn (by diff.)	0.035	....	0.035	0	71	....	17 400	17 600	49 000	55	21 000	16 000	15 000	0.31
	0.035	21	....	0	....	73.5	54 000	53 000	64 500	18	27 300	16 700	16 000	0.25
	0.035	37	....	0	....	81.5	65 000	60 000	75 500	7	28 100	19 500	19 000	0.25
	0.035	37	....	45	....	81.5	63 500	57 000	73 800	7	28 400	19 500	18 500	0.25
	0.035	37	....	90	....	81.5	65 500	61 500	76 300	7	30 000	19 200	19 000	0.25
Tin Bronze (1.44 per cent Sn) 98.51 Cu, 1.44 Sn, 0.014 P, 0.09 Zn, 0.001 Pb, 0.002 Fe	....	60	....	0	....	78	64 500	63 200	68 900	2.5	27 200	19 100	19 000	0.28
		75	....	0	....	82	67 500	66 000	73 600	2	27 000	20 500	20 500	0.28
Tin-Bearing Red Brass (1.00 per cent Sn) 85.27 Cu, 1.00 Sn, 0.001 Pb, 0.001 Fe, 13.73 Zn (by diff.)	....	60	....	0	....	87	74 000	64 200	83 500	2	26 800	20 000	20 000	0.24
Phosphor Bronze, 5 per cent (A) 95.38 Cu, 4. Sn, 0.23 P, 0.001 Pb, 0.002 Fe	....	60	....	0	....	93.5	87 500	72 000	95 000	3.5	29 000	20 000	20 000	0.21

with angle of applied stress referred to the rolling direction are illustrated. The work also deals with the effects of additions of tin to copper and red brass and of lead to high brass.

Considerable work has been done in the past to determine the relative suitability of various materials for spring applications on the basis of their fatigue properties. In 1929 Townsend and Greenall (**1**)[2] found that the fatigue strength at 100,000,000 cycles of two alpha brasses, three special brasses, nickel silver, phosphor bronze, and silicon bronze, ranged from 12 to 36 per cent of their tensile strengths. The results indicated that there is no general correlation between fatigue strength and tensile strength and that the increase in fatigue strength with cold work is not proportional to the increase in tensile strength. The transcrystalline nature of fatigue failures was illustrated. This work was supplemented in 1937 by Greenall and Gohn (**2**) in a paper which included a greater number of alloys and a more thorough investigation of the effect of temper. Fatigue properties were found to be affected by grain direction only when directional properties were evident in the microstructure. Improved fatigue characteristics of a nickel-silver were associated with small grain size. Polishing the specimen surfaces increased fatigue strength. Plated finishes adversely affected fatigue properties if the base metal surface was not properly prepared.

In 1937 Price and Bailey (**3**) showed that for strip finished with a 50 per cent reduction by cold rolling, phosphor bronzes with about 4.5 per cent and 7.5 per cent tin had higher fatigue properties than a 3 per cent silicon bronze and a tin-bearing copper-nickel alloy. The rather high values of fatigue strength, ranging from 27,000 psi. to 36,000 psi., at 50 million cycles were attributed to the fact that the tests had been made on polished specimens. Their results indicated that fatigue strengths derived from specimens in the commercially rolled condition would be considerably lower.

The fatigue properties of beryllium-copper strip in various tempers and stages of heat treatment were described by Gohn and Arnold (**4**) in 1946. A broad study of fatigue properties and their relation to other physical properties showed that for given tempers and heat treatments the other properties varied widely with variations in composition but that fatigue properties did not change. An increase in fatigue strength of about 15 per cent was effected in all tempers by heat treatment at 600 F. Maximum fatigue properties depended on adequate age-hardening and were not adversely affected by overaging. Improvements in fatigue strength were effected by surface treatments such as buffing or polishing.

Gohn and Ellis (**5**) in 1947 described fatigue characteristics of copper-nickel-zinc and phosphor bronze strip for conditions of unsymmetrical loading. The results obtained for both materials showed that the limiting range of alternating stress was constant and independent of the mean stress, provided the maximum stress did not exceed the yield stress.

MATERIALS

Chemical compositions and tempers of the materials included in this investigation are listed in Table I. All materials were processed in the form of strip rolled to a final thickness of 0.032 in. and then roll flattened. Annealing temperatures were carefully controlled to provide the desired grain sizes, particularly on the ready-to-finish gages. The materials tested in annealed tempers were made from strip previously rolled 37 per cent to 0.032 in. Specimens were milled from the hard rolled and flattened stock before the final annealing operation.

---

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 732.

TESTING EQUIPMENT AND PROCEDURE

Two fatigue testing machines of the type developed by the Bell Telephone Laboratories (1) for testing sheet metal were used in this investigation. Forty specimens in each machine are deflected alternately to both sides of a neutral position as cantilever beams held at one end and moved at the other end by reciprocating arms. These arms are motor driven through a shaft fitted with adjustable eccentricities at both ends. The specimens are thus stressed in alternate tension and compression by reversed bending, the magnitude of the stress depending on the deflection and the modulus of elasticity of the test material. The actual stresses were determined by independent, static, load-deflection tests on all the materials.

Specimens were milled by a form cutter from rectangular blanks, $\frac{1}{2}$ by 6 in., of the roll flattened strip. The specimen dimensions were the same as those used by Townsend and Greenall (1) and Price and Bailey (3). A region of uniform stress about $\frac{3}{8}$ in. long is incorporated in the design of the specimen by means of a taper in its width which effects a uniform bending moment in the fatigue test. Fillets of 2-in. radius connect the tapered section to the adjacent sections of the specimen.

Because the specimens were cross-milled with respect to the neutral plane, it was necessary to remove the tool marks which might otherwise act as stress raisers in the fatigue test. This was done by hand filing in a manner which did not change the contour of the edges. The rolled surfaces of the specimens were not altered since it was desired to test the materials with a commercially rolled finish. On the basis of other work (2, 3), it is probable that higher values of fatigue strength would have been obtained in this investigation if the specimens had been highly polished.

The fatigue testing machines were operated at a speed of approximately 900 cycles per min. Deflections of the reciprocating arms were adjusted to the nearest 0.0005 in. Allowance was made for differences between the static and dynamic deflections of each reciprocating arm at the extreme positions of its stroke. The general practice was to test four specimens of each material at a given deflection. Tests were run to a maximum of 100,000,000 cycles.

DISCUSSION OF RESULTS

Results of hardness, tension, and fatigue tests are summarized in Table I. The *S-N* curves relating stress and number of cycles to failure for all the materials tested are shown in Figs. 1 to 12. Correlations of fatigue strength, tensile strength, composition, temper and angle with rolling direction are shown in Figs. 13 to 18.

Usually the average number of cycles to failure for a group of specimens at a given deflection was used in plotting the *S-N* curves. In some instances, most frequently near the flat portion of the *S-N* curves where the spread between cycles to failure for duplicate specimens was great, the individual values were plotted. Points on the *S-N* curves which have one or more small lines above or below them refer to such individual specimens, the number of lines indicating the number of specimens. Arrows attached to points indicate that failure did not occur at the indicated number of cycles. In a few cases the *S-N* curves were not clearly defined by the distribution of points in the lower range of stress. The flat portion of such curves was gen-

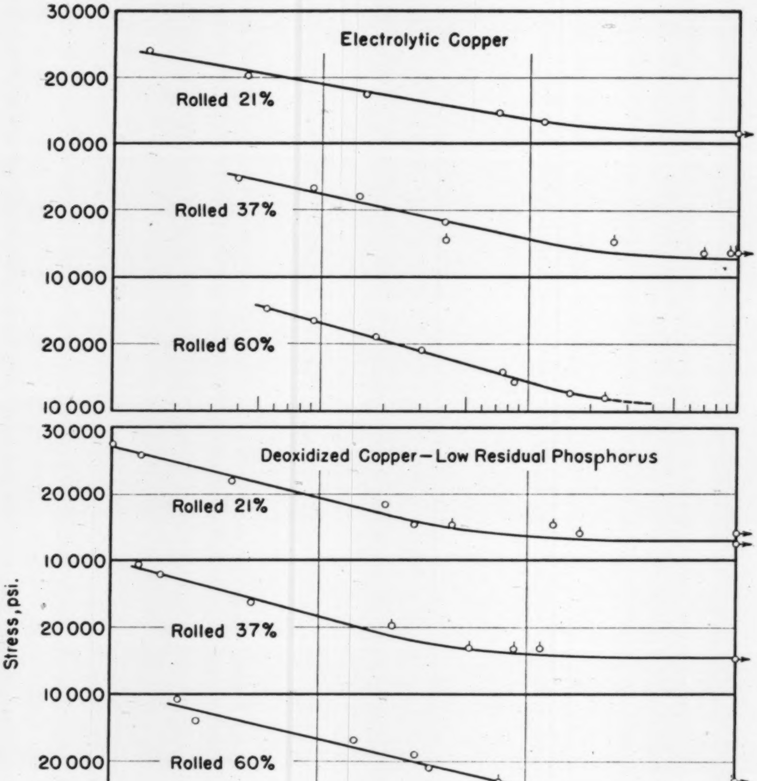


FIG. 1.—*S-N* Curves for Electrolytic Tough Pitch Copper and Deoxidized Coppers with 0.008 per cent and 0.027 per cent Residual Phosphorous.

All specimens cut with length parallel to rolling direction.

erally drawn slightly below these points in order to avoid the possibility of exceeding conservative estimates of fatigue strength.

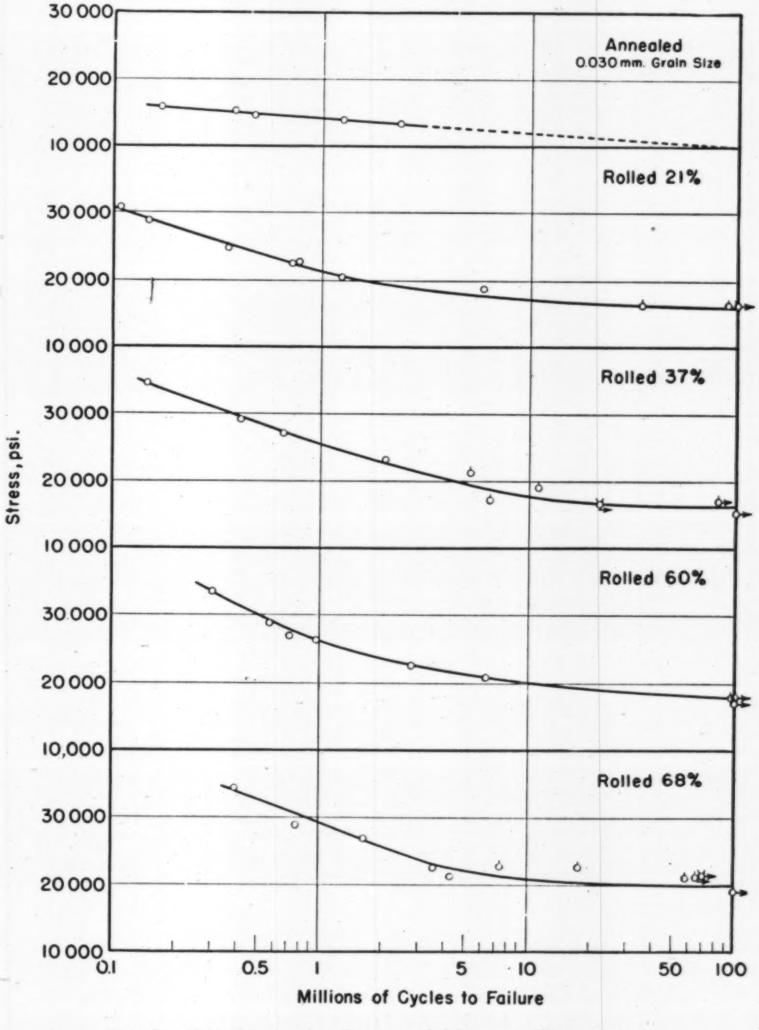


FIG. 2.—*S-N* Curves for Commercial Bronze, 90 per cent.
All specimens cut with length parallel to rolling direction.

Coppers:

The test results for the three types of copper are given in Figs. 1, 13, 14, 15, and 18. It is to be noted that the tensile strength increases steadily with degree of reduction up to 60 per cent and that, while fatigue strength at 100,000,000 cycles (the criterion of fatigue strength used throughout this discussion) shows a similar trend up to 37 per cent reduction, it then decreases at 60 per cent reduction. The value for the electrolytic tough pitch copper at 60 per cent reduction is actually less than for 21 per cent reduction. The deoxidized copper of 0.008 per cent phosphorus has essentially the same fatigue strength at 21 per cent and 60 per cent reductions. For the deoxidized copper of 0.027 per cent phosphorus content, the fatigue strength at 60 per cent reduction, while less than at 37 per cent reduction, is definitely greater than for 21 per cent reduction. The decrease of fatigue strength at the higher degree of rolling was encountered only for the three coppers. For the other materials which were tested, the general trend was for fatigue strength to increase when the reduction by rolling was increased.

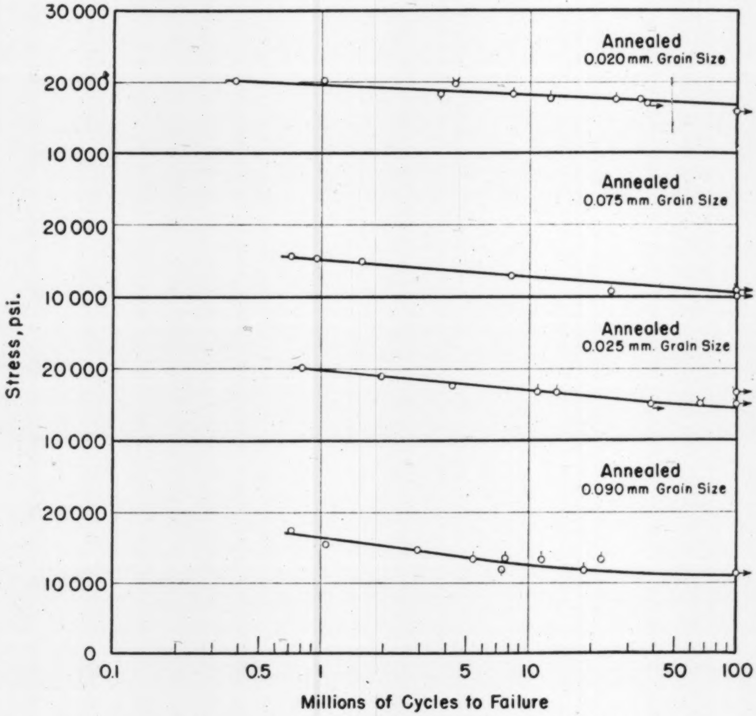


FIG. 3.—*S-N* Curves for Red Brass, 85 per cent—Annealed Tempers.

Materials of 0.020 mm. and 0.075 mm. grain sizes had 0.025 mm. nominal ready-to-finish grain size. Materials of 0.025 mm. and 0.090 mm. grain sizes had 0.075 mm. nominal ready-to-finish grain size. All specimens cut with length parallel to rolling direction.

As shown in Fig. 15, additions of 0.008 per cent and 0.027 per cent phosphorus increase the fatigue strength of copper, with the higher amount of phosphorus having the greater effect. The comparison is made by using the electrolytic tough pitch copper as the base material with no phosphorus present in it. Because of its oxygen content, this copper is perhaps a questionable standard of comparison, but discounting this factor, there appears to be a steady increase of fatigue strength with phosphorus content. This trend is not reflected in the relationships between tensile strength and phosphorus content—as rolled 37 per cent and 60 per cent, the deoxidized copper with 0.008 per cent phosphorus has the least strength of the three types of copper.

Copper-Zinc Alloys:

The results for commercial bronze, the 10 per cent zinc alloy, are given in Figs.

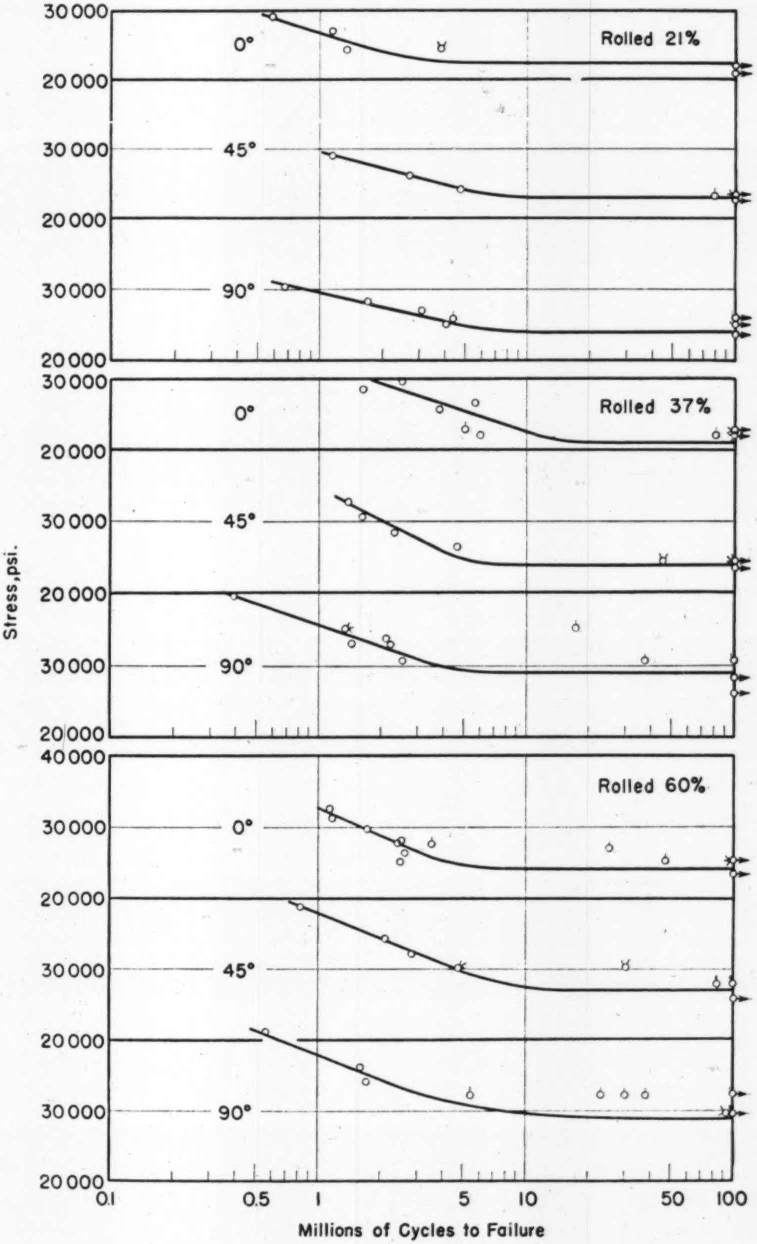


FIG. 4.—*S-N* Curves for Red Brass, 85 per cent—Hard Tempers, Rolled from 0.025 mm. Nominal Ready-to-Finish Grain Size.

Specimens cut with length parallel, 45 deg., and 90 deg. to rolling direction as indicated.

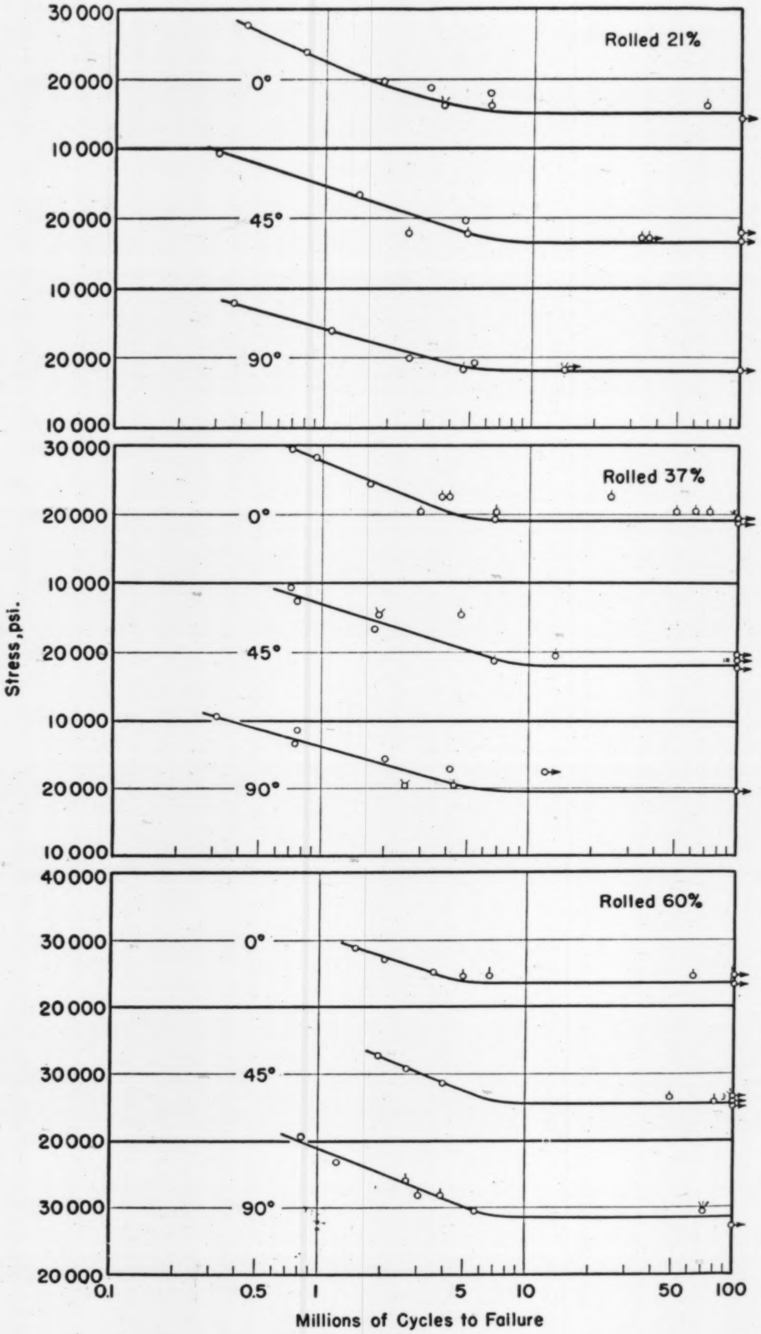


FIG. 5.—*S-N* Curves for Red Brass, 85 per cent—Hard Tempers, Rolled from 0.075 mm. Nominal Ready-to-Finish Grain Size.

Specimens cut with length parallel, 45 deg., and 90 deg. to rolling direction as indicated.

2, 13, 14, and 18. Both tensile strength and fatigue strength increase steadily with degree of reduction by rolling and, as may be ascertained in Fig. 18, there is a fairly constant relation between fatigue strength and tensile strength.

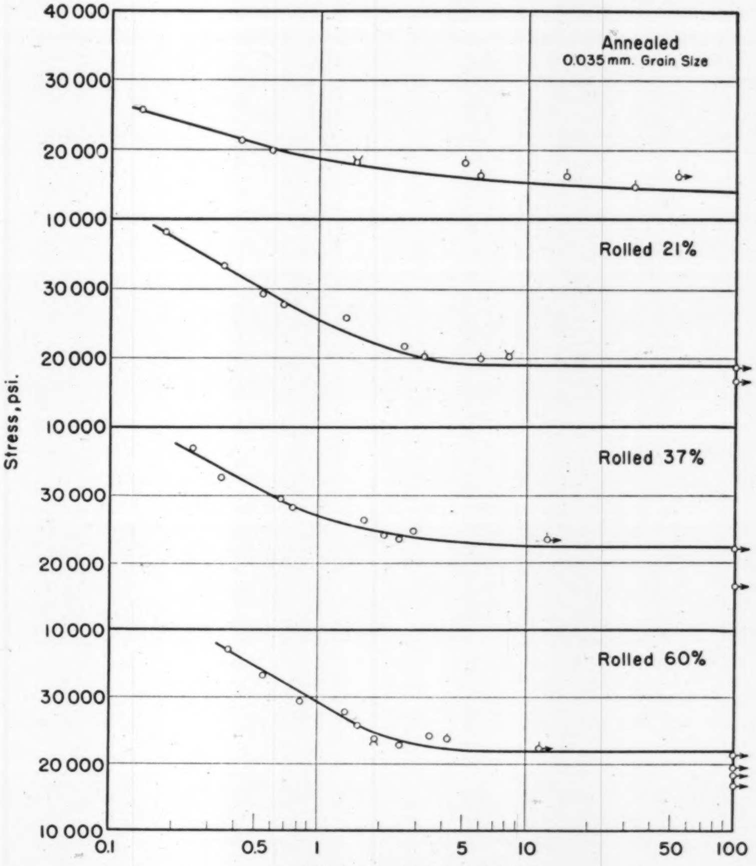


FIG. 6.—*S-N* Curves for Low Brass, 80 per cent.

All specimens cut with length parallel to rolling direction.

The red brass containing 15 per cent zinc was investigated in very considerable detail (Figs. 3, 4, 5, 13, 14, 16, and 18). Results were obtained for two orders of grain size for annealed material and it was found that the fatigue strength and the ratio of fatigue strength to tensile strength were greater for the finer grained material. A similar situation was found to prevail for material rolled 21 per cent after having been annealed to the two different grain sizes. For the 37 per cent and 60 per cent reductions, however, the ratio of fatigue strength to tensile strength appeared the same regardless of the previous grain size. For a given reduction, the finer grained material had higher fatigue strength, and this is directly associated with the higher tensile strength developed in rolling the finer grained material by any given amount.

Specimens of the rolled tempers of the

red brass were tested not only as cut parallel to the rolling direction but also at 45 deg. and 90 deg. to the rolling direction. As indicated in Fig. 16, there is a well-developed trend which shows that fatigue strength increases as the angle with the rolling direction increases from 0 to 90 deg. The trend is generally similar to the relationships which prevail between tensile strength and angle with the rolling direction. The relationship between fatigue strength and angle with rolling direction is more distinct than for tensile strength, inasmuch as the latter shows a tendency to have a minimum tensile strength at 45 deg. to the rolling direction for the lighter degrees of working.

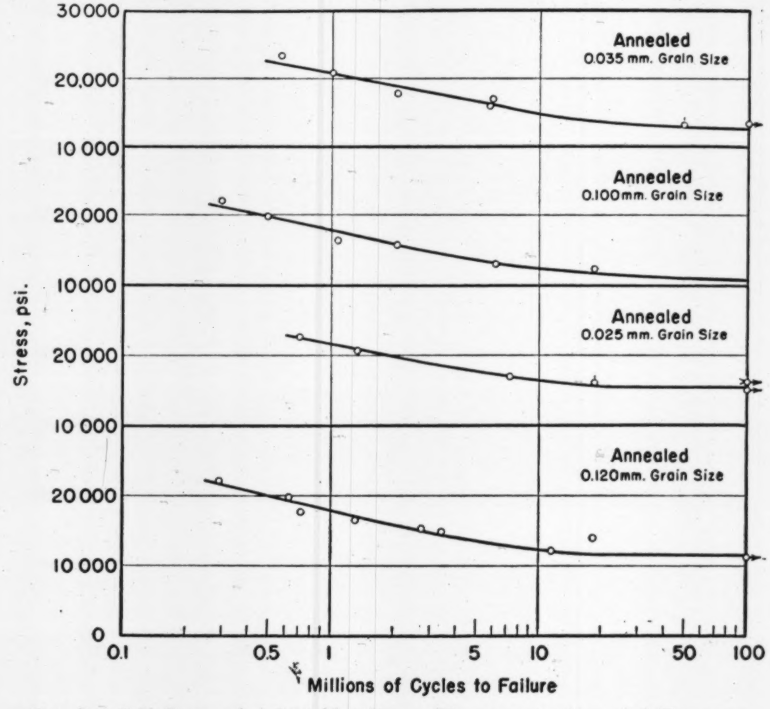


FIG. 7.—*S-N* Curves for Cartridge Brass, 70 per cent—Annealed Tempers.

Materials of 0.035 mm. and 0.100 mm. grain sizes had 0.025 mm. nominal ready-to-finish grain size. Materials o 0.025 mm. and 0.120 mm. grain sizes had 0.075 mm. nominal ready-to-finish grain size. All specimens cut with length parallel to rolling direction.

The results obtained for the low brass, containing 20 per cent zinc, are shown in Figs. 6, 13, 14, and 18. This alloy was tested only at 0 deg. with the rolling direction and here it was again found that fatigue strength increased with degree of reduction although there was little actual difference between 37 per cent and 60 per cent reduction. The ratio of fatigue strength to tensile strength is constant from 0 to 37 per cent reduction but is less at 60 per cent reduction.

The 70-30 brass (Figs. 7, 8, 9, 13, 14, 17, and 18) was investigated as extensively as the red brass. The test results show that the fatigue strength of annealed material of smaller grain is greater than that of larger grain. This dif-

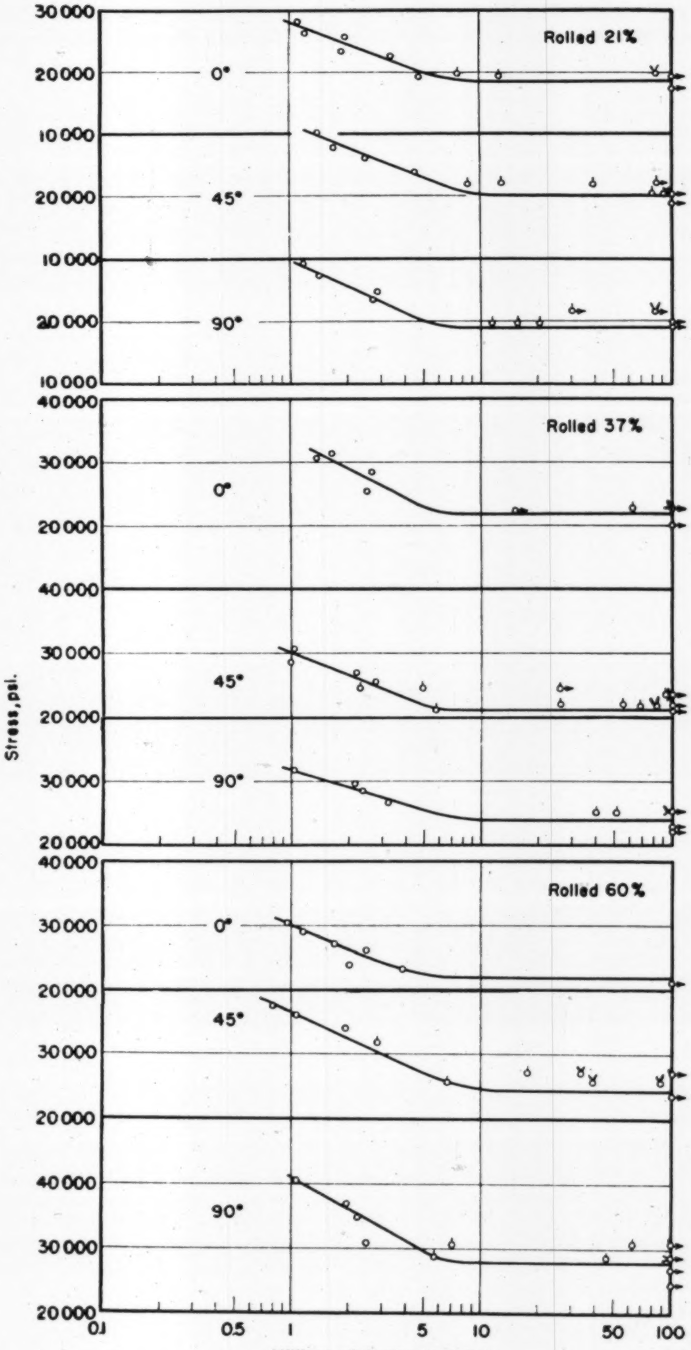


FIG. 8.—*S-N* Curves for Cartridge Brass, 70 per cent—Hard Tempers, Rolled from 0.025 mm. Nominal Ready-to-Finish Grain Size.

Specimens cut with length parallel, 45 deg., and 90 deg. to rolling direction as indicated.

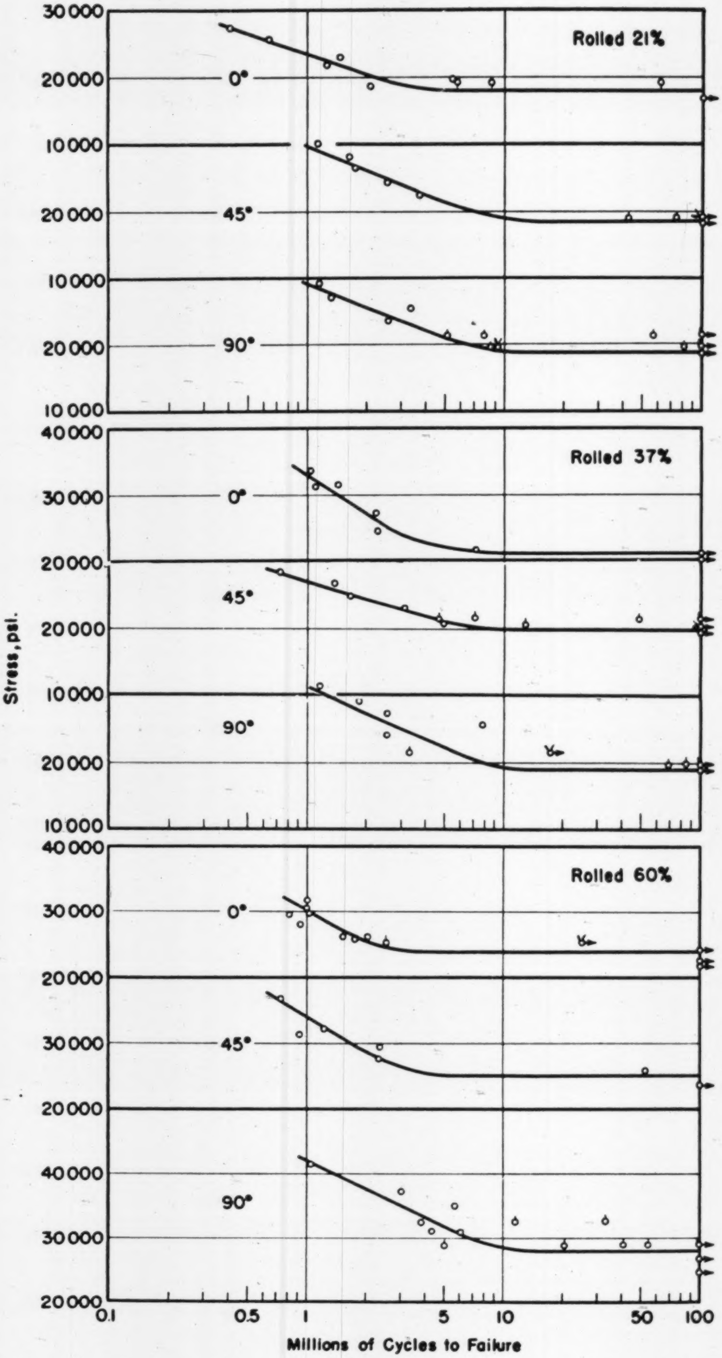


FIG. 9.—*S-N* Curves for Cartridge Brass, 70 per cent—Hard Tempers, Rolled from 0.075 mm. Nominal Ready-to-Finish Grain Size.

Specimens cut with length parallel, 45 deg., and 90 deg. to rolling direction as indicated.

ference due to grain size is less, however, than was observed in the case of the red brass.[7] The effect was also found in the rolled tempers for 21 per cent and 37 per cent reductions, but was actually reversed at 60 per cent reduction although the difference in fatigue strength for the two orders of previous grain size was very small. The ratio of fatigue strength to tensile strength is essentially constant from 0 to 37 per cent reduction, as shown in Fig. 18, and this again indicates a proportional increase in fatigue strength with tensile strength as the reduction by cold rolling becomes greater. The leveling off of the fatigue strength—tensile strength relationship in Fig. 18 reflects the marginal increase of fatigue strength at the most severe reduction.

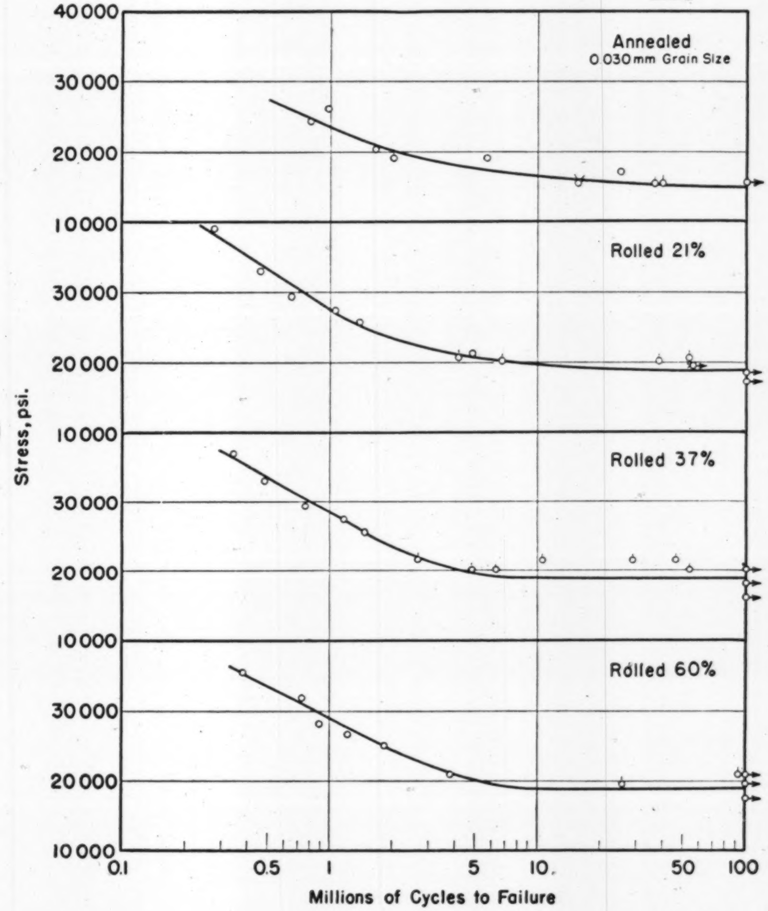


FIG. 10.—*S-N* Curves for High Brass, 65 per cent.

All specimens cut with length parallel to rolling direction.

The 70-30 brass in the rolled tempers was subjected to tests at 45 deg. and 90 deg. to the rolling direction as well as parallel to the rolling direction. The trends with respect to directionality are similar to those which have been discussed for the red brass, but are less well defined. Figure 17 shows a distinct trend of increasing fatigue strength with increasing angle to the rolling direction for the material as rolled 60 per cent. The lighter reductions are less positive but the general indication is one of slightly increasing fatigue strength as the angle to the rolling direction increases from 0 to 90 deg. It is to be observed that, while tensile strength of the alloy as

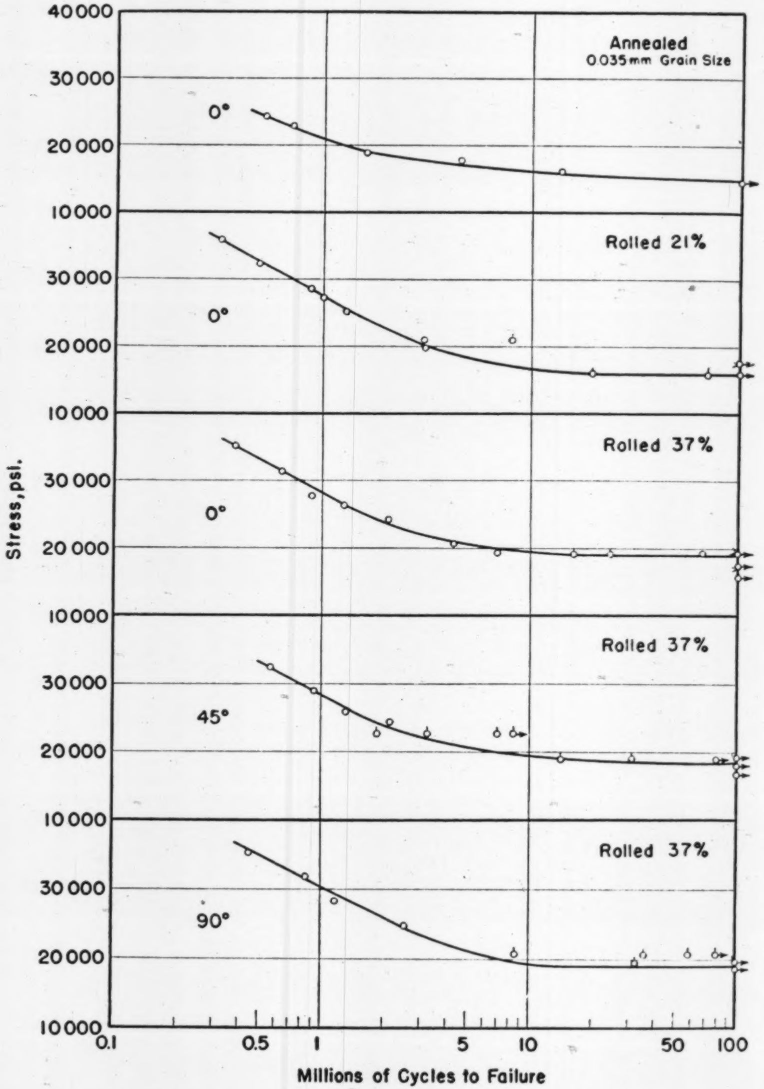


FIG. 11.—*S-N* Curves for High-Leaded Brass.

Specimens cut with length parallel, 45 deg., and 90 deg. to rolling direction as indicated.

rolled 60 per cent increases with angle to the rolling direction, there are minimum values of tensile strength at 45 deg. for the material as rolled 21 per cent and 37 per cent. With one exception, tensile strength is maximum at 90 deg. for these materials.

The brass containing 35 per cent zinc was investigated only parallel to the rolling direction. Results obtained for this alloy are shown in Figs. 10, 13, 14, and 18. While the tensile strength of this material increased steadily from the annealed condition to 60 per cent cold reduction, the fatigue strength increased only up to 21 per cent reduction and then it remained constant to 60 per cent reduction. Thus the ratio of fatigue strength to tensile strength can be considered fairly constant only to 21 per cent reduction and it decreases thereafter.

The general effect of zinc content on tensile and fatigue properties is summarized in Fig. 14, which contains data

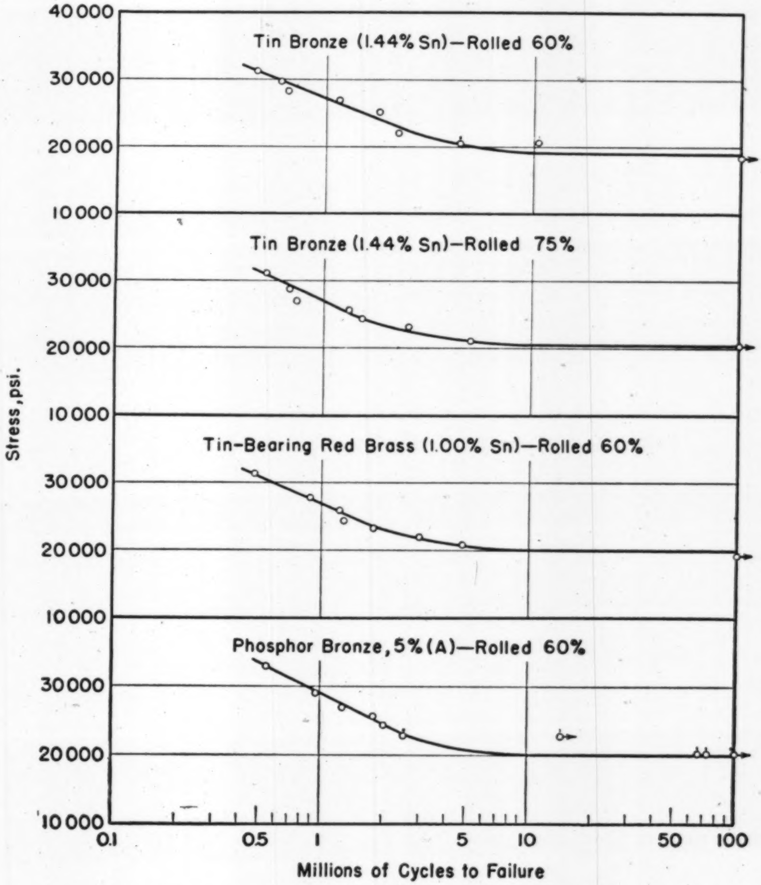


FIG. 12.—*S-N* Curves for Tin Bronze (1.44 per cent Sn), Tin-Bearing Red Brass (1.00 per cent Sn), and Phosphor Bronze, 5 per cent.

All specimens cut with length parallel to rolling direction.

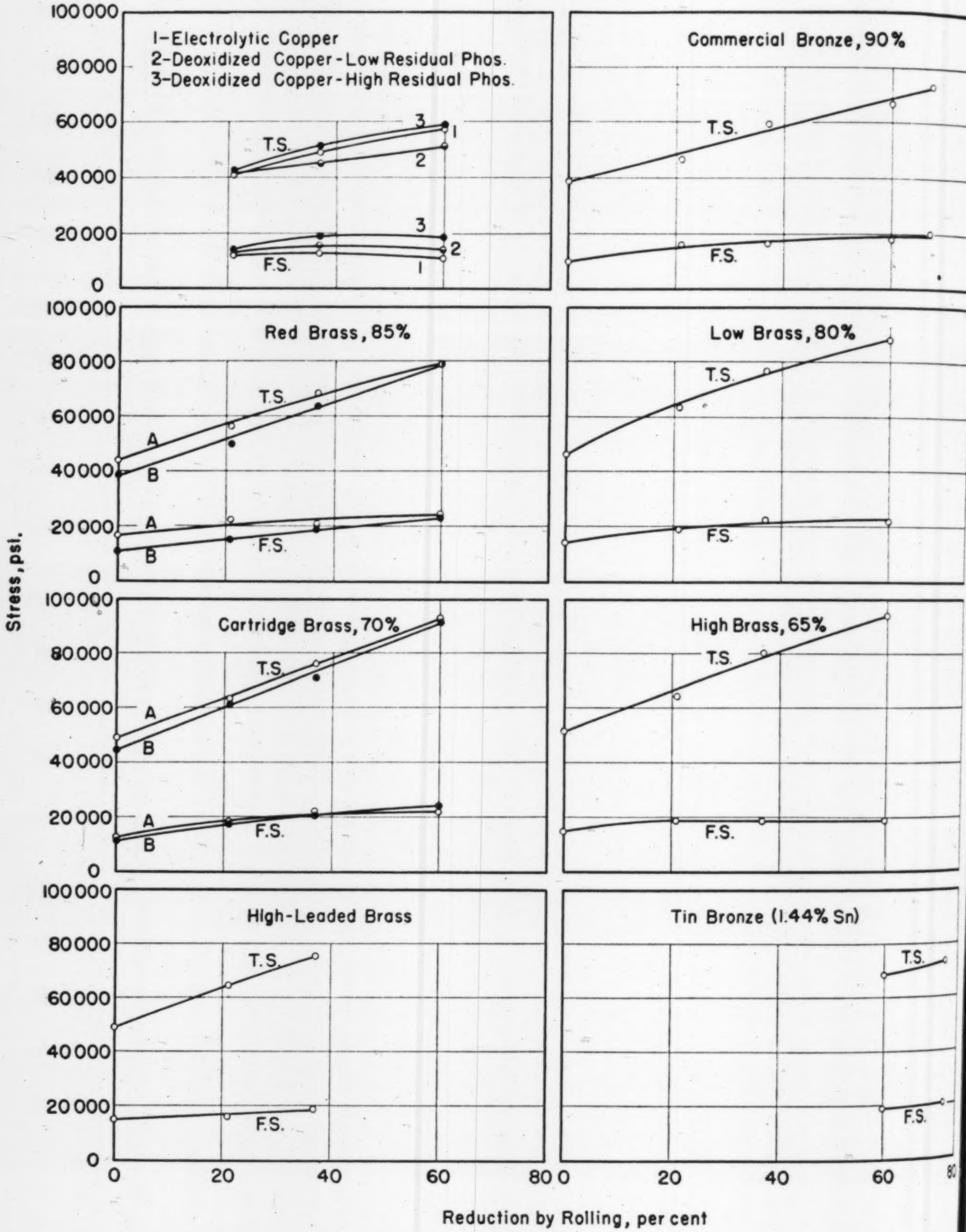


FIG. 13.—Relationship Between Tensile Strength (T.S.) and Fatigue Strength at 100,000,000 Cycles (F.S.) and Degree of Reduction by Rolling.

Specimens cut parallel to rolling direction.
Curve A—Materials of 0.025 mm. nominal ready-to-finish grain size.
Curve B—Materials of 0.075 mm. nominal ready-to-finish grain size.

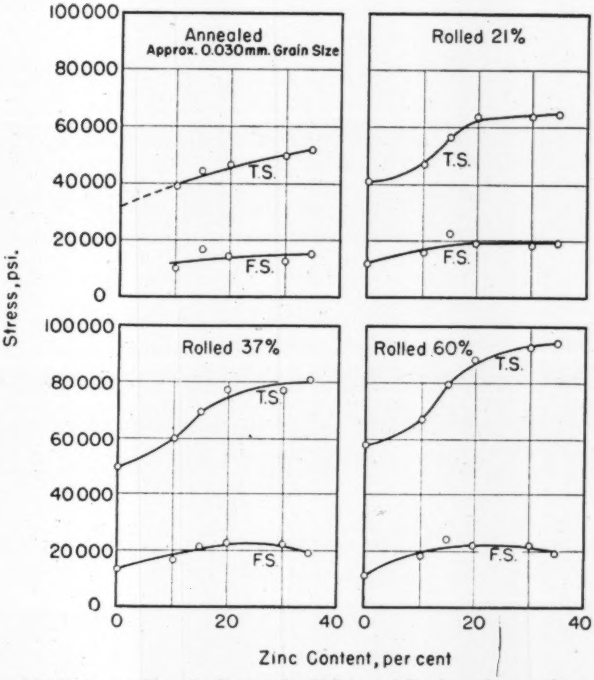


FIG. 14.—Relationship Between Tensile Strength (T.S.) and Fatigue Strength at 100,000,000 Cycles (F.S.) and Zinc Content of Copper-Zinc Alloys.

Specimens cut parallel to rolling direction.

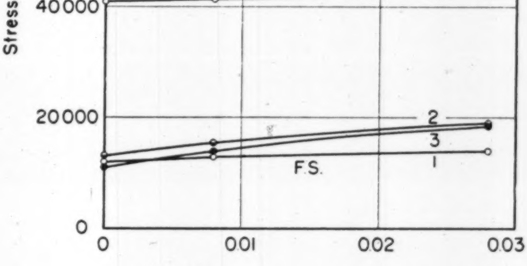


Fig. 15.—Relationships Between Tensile Strength (T.S.) and Fatigue Strength at 100,000,000 Cycles (F.S.) and Phosphorous Content of Copper.

Specimens cut parallel to rolling direction.

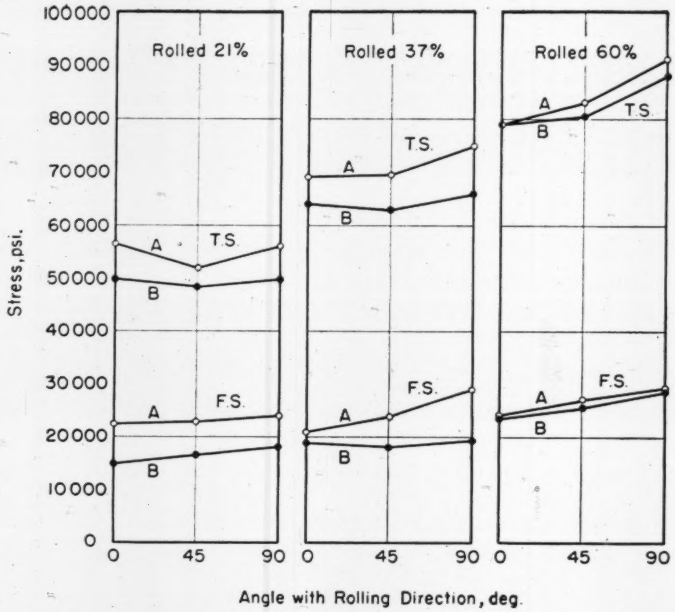


FIG. 16.—Relationships Between Tensile Strength (T.S.) and Fatigue Strength at 100,000,000 Cycles (F.S.) and Angle with Rolling Direction for Red Brass, 85 per cent.

Curve A—Materials of 0.025 mm. nominal ready-to-finish grain size.
Curve B—Materials of 0.075 mm. nominal ready-to-finish grain size.

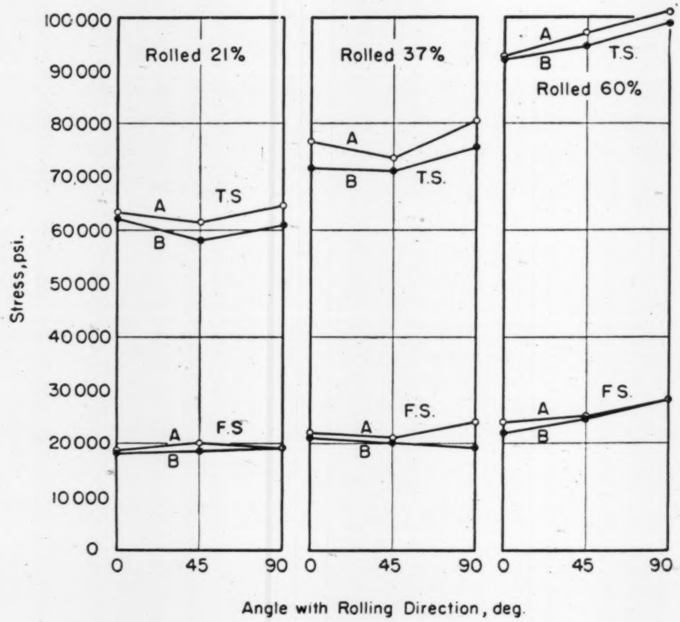


FIG. 17.—Relationships Between Tensile Strength (T.S.) and Fatigue Strength at 100,000,000 Cycles (F.S.) and Angle with Rolling Direction for Cartridge Brass, 70 per cent.

Curve A—Materials of 0.025 mm. nominal ready-to-finish grain size.
Curve B—Materials of 0.075 mm. nominal ready-to-finish grain size.

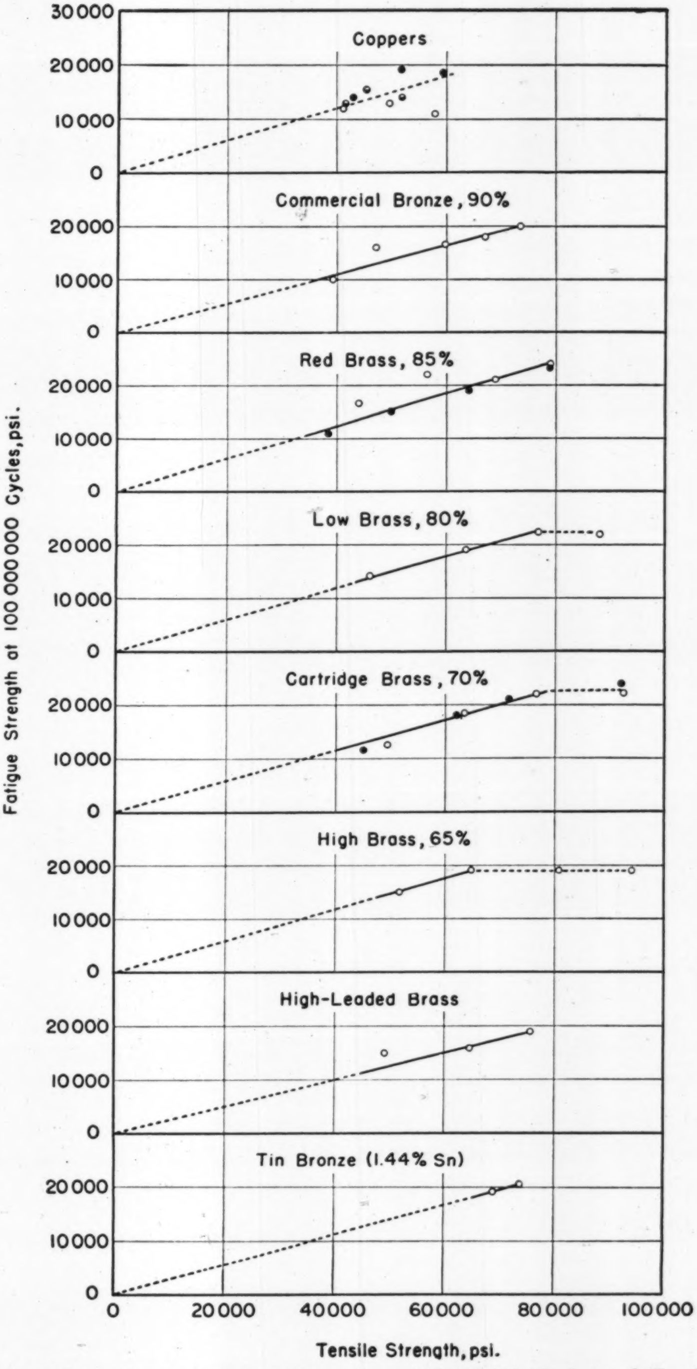


FIG. 18.—Relationships Between Fatigue Strength at 100,000,000 Cycles and Tensile Strength.

Specimens cut parallel to rolling direction. For the coppers, points indicated by open, half open-half solid, and solid circles refer to electrolytic tough pitch copper and deoxidized coppers of 0.008 per cent and 0.027 per cent phosphorus contents, respectively. For red brass, 85 per cent, and cartridge brass, 70 per cent, points indicated by open and solid circles refer to 0.025 mm. and 0.075 mm. nominal ready-to-finish grain sizes, respectively.

for strip rolled 21, 37, and 60 per cent from a nominal grain size of 0.030 mm. and also as annealed to a grain size of 0.030 mm. In the annealed condition, tensile strength increases steadily with zinc content up to 35 per cent zinc, as is well known, and the fatigue strength shows a slight increase from 10 per cent to 35 per cent zinc. As rolled 21 per cent, 37 per cent, and 60 per cent, the tensile strength increases considerably up to a zinc content of about 20 per cent and only slightly more for higher zinc contents up to 35 per cent.

For the hard-rolled tempers, fatigue strength increases with zinc content to substantially constant values throughout the range between about 15 per cent and 35 per cent zinc. Three of the four values of fatigue strength for the 15 per cent zinc alloy are higher than the general trends for zinc content shown in Fig. 14. There is some indication of a slight lowering of fatigue strength at 35 per cent zinc content for the 37 per cent and 60 per cent reductions.

A few of the materials of the cartridge brass and high brass compositions in this investigation were quite similar to some which were included in the work of Greenall and Gohn (2). It is interesting to note that, in general, the results obtained in the two investigations for materials of similar composition and treatment are in good agreement.

Lead in High Brass:

The results for the high-leaded brass strip, containing 2 per cent lead, are given in Figs. 11, 13, and 18. As tested parallel to the rolling direction, its tensile strength was virtually the same as for the cartridge brass and the high brass, at least up to 37 per cent reduction. Its fatigue strength is virtually the same as for high brass, the lead-free brass of similar copper and zinc contents. The alloy as rolled 37 per cent was tested at 0, 45, and 90 deg. with the rolling direction, and the fatigue strength was found to be the same at all three directions.

Tin in Copper and Brass:

The copper-tin alloy containing 1.44 per cent tin was tested as rolled 60 per cent and 75 per cent. Results are shown in Figs. 12, 13, and 18. The fatigue strength as rolled 60 per cent is 19,000 psi., whereas that of electrolytic tough pitch copper is only 11,000 psi. and that of deoxidized copper of 0.008 per cent phosphorus content is 14,000 psi. Its fatigue strength is not significantly better than that of deoxidized copper of 0.027 per cent phosphorus content in the same temper. The further rolling to 75 per cent reduction produces a slight increase in fatigue strength to 20,000 psi., which is in accord with the further increase in tensile strength. The grade A phosphor bronze (Fig. 12) as rolled 60 per cent was found to have a fatigue strength of 20,000 psi., which is no better than that developed by any of the brasses in the same temper. The reputed superiority of phosphor bronze strip as a spring material is probably associated with other characteristics such as high elasticity and freedom from relaxation rather than simple S-N relationships.

The fatigue strength at 100,000,000 cycles of 20,000 psi. for tin-bearing red brass (Fig. 12), rolled 60 per cent, shows that the addition of tin to red brass in strip form appears to effect no improvement in fatigue characteristics of the binary alloy. This differs from the effect on fatigue properties of tin additions to simple brasses in the form of small diameter wire. The authors showed in a previous paper (6) on fatigue properties of 0.072-in. diameter wire that additions of about $\frac{3}{4}$ to 1 per cent tin to 70-30 and 80-20 copper-zinc alloys, drawn 60 per cent and 80 per cent, improved both fatigue properties and tensile strength. Fatigue strength at 100,000,000 cycles was raised an average of 22 per cent and tensile strength an average of 12 per cent. The fatigue strengths of grade A phosphor bronze and a low (1.20 per cent) tin bronze in that investigation exceeded by about 10,000 psi. the fatigue strengths of corresponding alloys in the present work. The higher fatigue properties are associated with wire and the lower properties with strip of equal reductions. Differences in the nature of work hardening and surface quality effected by drawing and rolling may account for this behavior.

The difference in fatigue properties of wire and strip of the same composition and degree of reduction seems to be characteristic of certain alloys only. The simple brasses have similar properties in both forms. Values of fatigue strength for those brasses which are included in both the present investigation on strip and the earlier work (6) by the authors on wire are essentially equal for corresponding tempers.

Fatigue Strength-Tensile Strength Relationship:

The variation of fatigue strength at 100,000,000 cycles with tensile strength for most of the materials tested is shown in Fig. 18. Excluding some of the materials whose tensile strengths exceeded about 70,000 psi., there is a fairly linear relation between fatigue strength and tensile strength for the copper-zinc alloys from 10 per cent to 35 per cent zinc. It has already been indicated that the test results for the three types of copper do not follow such a relationship. Considering all of the materials in the investigation, fatigue strength ranges from 19 per cent to 44 per cent of tensile strength. On the basis of the linear relationships drawn in Fig. 18 for the copper-zinc alloys, fatigue strength is about 28 per cent (plus or minus 2 per cent) of tensile strength if the latter does not exceed approximately 70,000 psi.

SUMMARY AND CONCLUSIONS

The fatigue properties of three types of copper, five copper-zinc alloys, and four other copper-base alloys in strip form were determined by reversed bending tests. It was found that additions of phosphorus up to at least 0.027 per cent increase the fatigue strength of copper and that reduction of 37 per cent by rolling gives higher fatigue properties than reductions of 21 and 60 per cent.

Fatigue strength of the brasses increases with zinc content to a maximum near 15 per cent zinc and does not change significantly with increasing zinc content up to 35 per cent zinc. As a minor exception, the fatigue strength of material rolled 37 per cent or 60 per cent decreases slightly at a zinc content of 35 per cent. Some of the brasses included in this investigation show a fairly continuous increase in fatigue strength with increasing degree of reduction up to at least 60 per cent. The fatigue strength of the other brasses is increased by moderate cold working and remains fairly constant with further cold working up to at least 60 per cent reduction. Unlike the coppers, there is no decrease in fatigue strength at a higher reduction, such as 60 per cent, for any of the brasses. Excluding the coppers and the brasses of tensile strength greater than about 70,000 psi., fatigue strength increases proportionally with tensile strength. In general, fatigue strength of hard-rolled brass increases as the angle with the rolling direction increases.

The fatigue properties of annealed materials decrease with increasing grain size. Fatigue strength of hard-rolled materials decreases with increasing ready-to-finish grain size; the effect of ready-to-finish grain size diminishes with increasing degree of reduction.

The addition of 2 per cent lead to high brass has no significant effect on fatigue properties. The copper-tin alloy containing 1.44 per cent tin has higher fatigue properties than electrolytic tough pitch copper and deoxidized copper of 0.008 per cent phosphorus content. Grade A phosphor bronze, 5 per cent, has higher fatigue properties than any of the coppers tested. Fatigue characteristics of red brass are not altered by the addition of 1 per cent tin. All of these tin-bearing alloys in strip form have considerably lower fatigue properties than corresponding alloys in the form of small diameter wire.

Acknowledgments:

The authors acknowledge the interest of D. K. Crampton, Director of Research of the Chase Brass and Copper Co., in this fatigue testing program and the assistance of those who participated in various phases of the work. Permission to publish the results was given by the Chase Brass and Copper Co.

REFERENCES

(1) J. R. Townsend and C. H. Greenall, "Fatigue Studies of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 353 (1929).

(2) C. H. Greenall and G. R. Gohn, "Fatigue Properties of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 160 (1937).

(3) W. B. Price and R. W. Bailey, "Fatigue Properties of Five Cold Rolled Copper Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metal Div., Vol. 124, p. 271 (1937).

(4) G. R. Gohn and S. M. Arnold, "The Fatigue Properties of Beryllium-Copper Strip and Their Relation to Other Physical Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 741 (1946).

(5) G. R. Gohn and W. C. Ellis, "The Fatigue Characteristics of Copper-Nickel-Zinc and Phosphor Bronze Strip in Bending Under Conditions of Unsymmetrical Loading," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 713 (1947).

(6) H. L. Burghoff and A. I. Blank, "Fatigue Tests on Some Copper Alloys in Wire Form," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 774 (1943).

DISCUSSION

MR. G. R. GOHN[1] (*presented in written form*).—The authors are to be congratulated upon the completion of a meticulously planned and well-executed research program which contributes greatly to our knowledge of the fatigue characteristics of copper and copper alloys in strip form. Despite the fact that probably more springs are made from such materials than from all other materials combined, the number of investigations carried out in this field have been limited. Following the early pioneering work of Townsend and Greenall in 1929 (**1**)[2] and the extended studies of Greenall and Gohn in 1937 (**2**) no comprehensive studies on the fatigue characteristics of non-ferrous alloys in strip form have been undertaken until recently (**4**, **5**). In that interval, many changes in mill practice have been made which may also have an effect on the fatigue characteristics of the alloys. For this reason, if for no other, the studies of Messrs. Burghoff and Blank are of considerable interest. However, the authors' work is of even greater importance because they have included in their studies alloys and conditions not previously described by other investigators.

To my mind the most important conclusion resulting from the authors' work is the effect of grain size upon the fatigue strength of non-ferrous alloys in strip form, particularly those alloys subjected to reduction by cold rolling. The writer in 1937 (**2**) found evidence of this effect in his studies on nickel-silver strip and concluded that by the use of material having a small grain size improvement in the fatigue properties could be effected. This improvement in fatigue strength resulting from the use of fine grain size material was also observed in the writer's studies on beryllium copper strip as reported before this society at the 1946 Annual Meeting (**4**). It is gratifying, therefore, to find that the authors have studied the effect of grain size on fatigue properties so thoroughly and arrived at a similar conclusion. From their work it is quite evident that cold-rolled copper and copper alloys intended for spring applications should have a ready-to-finish grain size in the range of 0.020 to 0.025 mm. This important finding should, in my opinion, be put into mill practice at an early date and merits consideration by A.S.T.M. Committee B-5 for possible inclusion in the various specifications on strip materials under its jurisdiction.

In studies involving the determination of more than 140 *S-N* diagrams for 23 different non-ferrous alloys in various tempers and surface treatments, Greenall and Gohn (**2**) found that the ratio between fatigue strength at 10^8 cycles of reversed stress varied 14 to 40 per cent. This range compares very favorably with the authors' findings of 19 to 44 per cent for the alloys which they studied. The close agreement in the fatigue strength determinations of alloys common both to the authors' studies and those of Greenall and Gohn should also be noted. As shown in the accompanying Table II in no case does the difference exceed 2000 psi. or 10 per cent.

---

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to the authors' paper, see p. 732.

The authors point out that the fatigue strength of grade A phosphor bronze is not materially different from that of the copper-zinc alloys. This agrees with the earlier findings of Greenall and Gohn (2). However, in material obtained recently from two different sources, we have found that the fatigue strength of spring temper (60 per cent reduction) is in excess of 30,000 psi. at 10^8 cycles of reversed stress. We wonder whether this increase in fatigue strength indicates changing mill practices, particularly the use of a smaller ready-to-finish grain size or if the authors could otherwise account for this material improvement in fatigue properties. The composition of the alloys recently tested is almost identical with that reported by the authors for the grade A phosphor bronze which they studied. As stated by the authors this material is widely used for spring applications not only because of its good fatigue properties but also because of its excellent elastic properties, particularly its high yield strength at 0.01 per cent offset.

TABLE II.—COMPARISON OF FATIGUE TEST DATA.

Material	Temper, per cent reduction by cold rolling	Thickness, in.	Angle With Rolling Direction	Fatigue Strength, psi., at 10^8 cycles of reversed stress as determined by	
				Burghoff and Blank	Greenall and Gohn
Electrolytic Copper	21	0.032	Parallel	12 000	...
	21	0.024	Parallel	...	13 000
High Brass	0	0.032	Parallel	15 000	...
(Rolling Series)	0	0.024	Parallel	...	15 000
(Commercial)	0	0.024	Parallel	...	13 500
	37	0.032	Parallel	19 000	...
(Rolling Series)	37	0.024	Parallel	...	19 500
(Commercial)	37	0.024	Parallel	...	17 500
	60	0.032	Parallel	19 000	...
(Rolling Series)	60	0.024	Parallel	...	20 000
(Commercial)	60	0.024	Parallel	...	17 500
Grade A Phosphor Bronze	60	0.032	Parallel	20 000	...
	60	0.024	Parallel	...	22 000

The authors' findings that cold-worked copper-zinc alloys have essentially the same fatigue strength for comparable tempers for alloys varying from 15 to 35 per cent in zinc content are quite important as are their findings that the addition of 2 per cent of lead to high brass has no significant effect on fatigue properties. The importance of the work by Messrs. Burghoff and Blank will be appreciated more as the paper is studied in detail.

MR. JOHN A. BENNETT[3] (*presented in written form*).—The authors are to be congratulated on this paper which provides a large amount of valuable information on the fatigue properties of copper alloys.

The copper alloys are frequently used in the form of thin strip less than 0.01 in. thick which are difficult to test in bending fatigue because of the large deflection required to produce the desired stress. Consequently data are lacking for such thin material, and it is doubtful that data from tests on thicker material are accurately applicable to thin stock.

When this problem came up in connection with an investigation of the effect of casting variables on properties of bronze, a fixture was designed for testing thin strip which may be of general interest. The fixture, shown in the accompanying Fig. 19, is attached to a Krouse sheet bending fatigue machine and is designed to load the specimen as a pin-ended column. The specimen is of rectangular shape 1 by $\frac{1}{2}$ in. and is held in grooves in the lower fixed support and in the dummy specimen above. The dummy specimen has a flexure plate support in the vise.

---

[3] Metallurgist, National Bureau of Standards, Washington, D. C.

In setting up the specimen, the deflection at the center is measured by means of a scale placed so as to indicate the distance from the line between the supporting grooves. In the test the minimum deflection is usually taken as $\frac{1}{2}$ mm. and the maximum is adjusted to give approximately the desired stress.

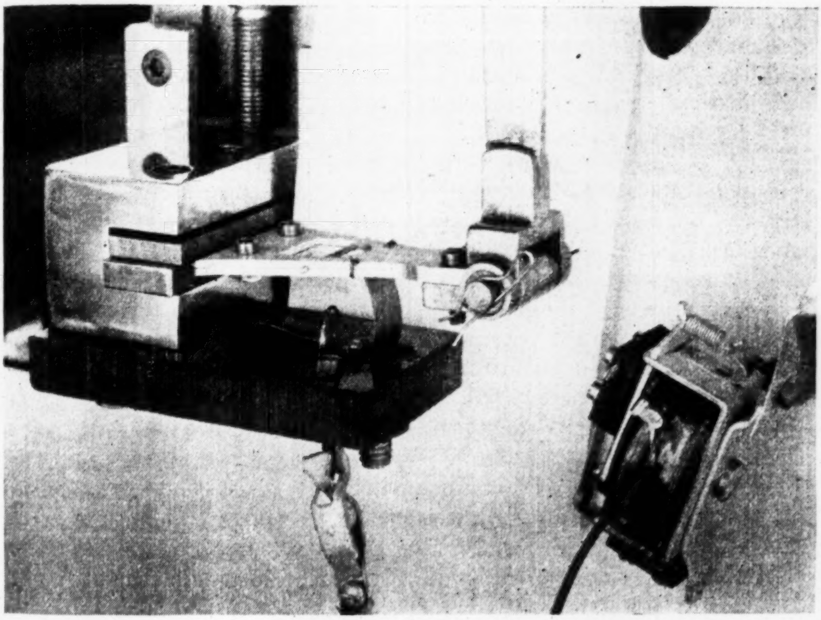


FIG. 19.—Fixture for Testing Thin Strip.

The maximum stress on the specimen may be calculated by either of two methods: first, from a measurement of the radius of curvature at the center of the specimen in cases where the elastic modulus of the material is known. The second method involves the determination of the bending moment at the center, from which the stress in the outer fibers can be calculated without knowing the elastic constants of the material. In order to determine the bending moment, the load required to produce the deflection to be used in the test is determined in a jig made for the purpose, then the product of this load and the deflection gives the bending moment.

The results of tests made with this fixture on 0.005 in. thick strip are reported in a paper now being prepared for publication by Mr. H. B. Gardner of the Metallurgical Division.

MR. BENNETT (*added orally*).—I would also like to ask the authors if they have any explanation for the effect of rolling direction on fatigue properties which is contrary to what might be expected?

MR. T. J. DOLAN.[4]—There have been several instances in recent years in which fatigue tests of cold-drawn copper wires have indicated a recrystallization occurring during the progress of the fatigue test. I wonder whether the authors have made a subsequent metallographic examination of any of these copper or copper-alloy specimens to determine whether they have also had such phenomena occurring?

---

[4] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

It seemed to me offhand that since the recrystallization temperatures are somewhat depressed by greater degrees of cold working, and since these temperatures are only a slight amount above room temperature, this might be one possible explanation why the materials which were rolled to the greatest extent did not show a corresponding increase in fatigue strength at the higher amounts of cold reduction. That is, if recrystallization occurs during repeated stressing (even though to a very minor extent and on a microscopic scale), the larger crystal size resulting is representative of a weaker material and might preclude the attainment of a higher fatigue strength.

MR. A. I. BLANK (*author's closure*).—The authors appreciate the interest which has been shown in this paper by Messrs. Gohn, Bennett and Dolan.

The close agreement of the results obtained in the present work with those reported by Greenall and Gohn (2) for similar materials was noted by the authors during the course of the investigation.

Mr. Gohn's reference to the fatigue strength of grade A phosphor bronze is of great interest because it brings up the matter of the effect of ready-to-finish grain size. The grain size of the phosphor bronze strip used in this investigation has been checked and found to be relatively large, about 0.120 mm., prior to the final rolling. On the basis of the marked effect of grain size found for other materials, it is reasonable to expect that a smaller grain size of the order of 0.025 mm. would yield an appreciably higher value than 20,000 psi. for the same degree of final reduction. The fatigue strength of 30,000 psi., which Mr. Gohn reported for two recent lots of phosphor bronze, is indicative of a small or moderate grain size.

Further examination of the 1.44 per cent tin bronze and of the tin-bearing red brass used in this investigation has shown that these materials had moderately large ready-to-finish grain sizes, about 0.060 to 0.080 mm. For small ready-to-finish grain sizes the fatigue properties of these alloys would probably be significantly higher than the listed values.

With regard to the effect of rolling direction on fatigue properties of hard-rolled strip mentioned by Mr. Bennett, it is assumed that the lower capacity for being plastically bent along the direction of rolling is the basis for questioning the reported increase in fatigue strength with increasing angle referred to the rolling direction. Actually this basis does not apply because the stresses in the fatigue tests were within the elastic range and therefore plastic behavior is not a suitable criterion. The relations between rolling direction and tensile and fatigue strengths are similar; that is, strength normal to the rolling direction is generally greater than that parallel to the rolling direction.

It seems unlikely that recrystallization on a microscopic scale, suggested by Mr. Dolan, can account for the slight decrease in fatigue strength of the coppers on rolling beyond a moderate degree of reduction. Although microscopic examination of the fractured specimens was not made, there is reason to believe that recrystallization did not occur during the fatigue tests at room temperature. No temperature rise in the fatigue specimens was noted during the course of the fatigue tests. The results of recrystallization studies which we have made on various types of copper indicate that many years would be required for any recrystallization to occur at or slightly above room temperature in commercial copper rolled 60 per cent. The recrystallization in copper at room temperature which has been observed by Cook and Richards[5] applies to copper rolled to a much higher degree of reduction.

---

[5] M. Cook and T. Ll. Richards, "The Self Annealing of Copper," *Journal*, Inst. Metals, Vol. 70, No. 4, pp. 159-173 (1944).

TENSILE, CREEP AND FATIGUE PROPERTIES AT ELEVATED TEMPERATURES OF SOME MAGNESIUM-BASE ALLOYS*

By John C. McDonald[1]

Some information has already been published on the properties of magnesium-base alloys at elevated temperatures (**1**, **2**, **3**, **4**).[2] There are not, however, comprehensive data readily usable by designers. The purpose of the work described in this paper was to obtain the desired information for the magnesium-base materials most likely to be used in such applications as engines. The following types of tests were considered necessary to evaluate properly the available materials: tensile properties at room and at elevated temperatures as a function of time of exposure at elevated temperatures; creep tests for various times and amounts of extension up to and including rupture; fatigue tests under various conditions of loading and stress concentration. With the results of such tests at hand the task of the designer should be much facilitated in selecting a material satisfactory for his operating conditions. The increasing use of magnesium in elevated temperature applications, such as engines, provided a powerful impetus for the prosecution of this program.

The program of tests to accomplish these objectives was started and has now been largely completed. Gaps in the data at a few spots have made it necessary to resort to estimation. Where this has been done, the fact is indicated on the figures or in the text. It is hoped that the publication of this information will be a useful guide in the application of magnesium to a number of different kinds of parts in operation at elevated temperature.

At the time this work was begun there was no indication that the properties of sheet at elevated temperature might be of great interest for certain types of applications. In consequence, the tests reported herein were carried out entirely on castings and forgings. A need now exists for a similar study on sheet alloys.

Materials

Previous work has shown that alloying elements added to magnesium differ markedly in their effect on properties at elevated temperature. This difference is most marked in the case of creep. In increasing order of potency in stopping creep, four of the elements studied in the past are as follows: aluminum, manganese, zinc, and cerium. Alloy composition may be varied considerably as long as any one of the above elements is retained as the major alloying constituent without greatly affecting the creep properties. The differences in static properties produced by the above elements are considerably smaller than is the case for the creep properties. This paper will treat of alloys containing aluminum, manganese, or cerium as the major constituents. Study of alloys in which zinc is the main component are still in a preliminary stage. Our current knowledge of them is embodied in the work of Leontis (**4**).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Assistant Technical Director, The Dow Chemical Co., Midland, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, p 753.

In order to keep the amount of work required within reason, not more than one alloy was selected for test from each group for a given method of fabrication. Two methods of fabrication were studied —sand castings and forgings. All materials were heat treated in such a way that a reasonable degree of stability at elevated temperatures was secured consistent with attainment of maximum properties at those temperatures.

TABLE I.—MATERIALS TESTED.

Alloy	Temper Designation	Solution Treatment	Aging Treatment	Form	Composition Range, per cent		Mn, per cent, min.	Ca per cent, max.	Ce, per cent[b]	Tensile Strength, 1000 psi.		Yield Strength, 0.2 per cent offset, 1000 psi.		Elongation, per cent	
					Al	Zn				Batch	Spec.[c] Min.	Batch	Spec.[c] Min.	Batch	Spec.[c] Min.
AZ63	HTS	730 F.—12 hr.	500 F.—4 hr	Sand casting	5.3 to 6.7	2.5 to 3.5	0.15	...	...	39	34	18	13	8	4
AZ80X	HTA	750 F.—12 hr.	350 F.—16 hr.	Forging	7.8 to 9.2	0.4 to 1.5	0.12	...	...	48	42	30	28	7	2
M1	*a*	...	700 F.—1 hr.[a]	Forging	...	...	1.2	0.3	...	34	...	19	...	12	...
EM61	HTA	1050 F.—16 hr.	400 F.—16 hr.	Sand casting	...	...	1.2	...	4.8 to 7.2	19	...	18	...	1	...
EM51	HTA	1050 F.—4 hr.	400 F.—16 hr.	Forging	...	...	1.2	...	3.8 to 6.2	38	...	29	...	5	...

[a] An annealing treatment
[b] Total rare earths—Ce added as the "Mischmetal" of commerce.
[c] Dow Chemical Company Specifications.

The materials selected are listed in Table I. The first two alloys, AZ63 and AZ80X, are representative of a high-aluminum type alloy in the cast and wrought condition, respectively. The behavior of AZ63 is representative of other alloys such as AZ92, A10, AZ91, and A8. The condition in which it was tested, heat treated and stabilized (HTS), was considered to be slightly more stable than the heat treated and aged temper (HTA). The latter temper is secured by a longer time of aging at a lower temperature. Other work has shown, however, that the tempers are substantially equivalent under the conditions of tests used in this work.

Of the various wrought alloys containing aluminum, AZ80X has the highest alloy content, the greatest strength and the highest hardness. Creep properties of alloys of lower aluminum content are quite similar.

The manganese-containing alloy, M1, is the only representative of its class used commercially. It is not used in the cast form for elevated temperature applications, so no tests were made.

The alloys in which cerium is the major alloying constituent also contain manganese. The purpose of this latter addition was the usual one in magnesium metallurgy, to enhance the corrosion stability of the material. Similar compositions were used commercially in Germany, although no heat treatment was applied. Commercial practice there was confined to forgings. Current practice in this country favors a composition without manganese for castings. It has been found that the large grain size produced by the presence of manganese increases the possibility of cracking in the mold. This effect was not observed in small simple castings, but arose immediately when larger, more complicated castings of the aircraft engine type were tried.

The effect of the manganese on mechanical properties is small. The alloy now being used, E6, is considered equivalent to the EM61 in serviceability. Laboratory data indicate that forgings may require less than 5 per cent cerium for the same level of properties at elevated temperature. Materials developed as the result of future studies along this line will be quite similar to the EM51 in respect to the properties reported in this paper.

There are no A.S.T.M. specifications for the particular materials and tempers covered in this paper. Minima specified in Table I are as currently used by The Dow Chemical Co.

The sand-casting alloys were tested in the form of separately cast test bars, bars cut from flat panels, and in one case, bars cut from a commercial casting, to which reference will be made later. Grain refinement was carried out on AZ63 by the usual superheating treatment. The grain of EM61 was refined by treating with zirconium in the form of a fused salt containing two parts $ZrCl_4$, and one part each of KCl and NaCl. The effect is comparable to that produced by not using manganese in the alloy. The latter procedure is much simpler, and as stated earlier, was adopted for production in the foundry of the large aircraft engine castings.

Forgings were prepared by flattening 4-in. diameter round extrusions in an hydraulic press. The axis of the extrusions was normal to the forging direction. Most of the tests were carried out on the material flattened to 1-in. thickness in one forging step. Fatigue tests of the plate bending type were carried out on the same material given a second forging step down to a thickness of $\frac{1}{4}$ in. Temperature of forging for AZ80X and M1 was 700 F.; for EM51, 1000 F. Heat treatments were carried out as shown in Table I.

The applicability of the data obtained to the performance of actual parts should be discussed. It is well known that the properties of such parts often depart from those of specimens fabricated under optimum conditions. Excluding obvious defects which are usually remediable, the chief remaining factor in the case of castings is grain size. This is considerably affected by the thickness of sections being cast and by methods of molten metal treatment. Variations in grain size exert a considerable effect on room temperature tensile properties. Their effect on fatigue and creep properties, however, has been shown to be much smaller, within the range of grain size considered likely to be encountered in commercial castings. Wrought metal likewise can vary greatly in room temperature properties depending upon the conditions of forging and the amount of reduction from the cast condition. The variations in tensile properties at elevated temperature, however, are less than those observed at room temperature. The effect on creep and fatigue is of a still smaller order.

METHOD OF TESTS

A detailed description of the method of tests has already been published and need not be repeated here (**1**, **2**). Tensile and creep testing were carried out exactly as described by Moore and McDonald (**1**). Notched bar rotating-beam fatigue tests were performed at a stress concentration factor of 2 only. Plate bending fatigue was done at a mean stress equal to zero (completely reversed stress). No notched bars were tested. Axial loading fatigue tests were carried out at a ratio of minimum to maximum tensile stress of $\frac{1}{4}$. A notch of stress concentration factor 2.4 was the only one studied.

Results

The test results are presented in a series of graphs. In general, each graph comprises a related series of curves. All test points are shown. The number of curves that could be put on one graph was necessarily limited in order to avoid confusion from overlapping of points and curves. As it is, some of the graphs contain more points and curves than would be desirable from the standpoint of maximum clarity. However, any further simplification would greatly increase the already large number of graphs required to present all the test data.

The effect of exposure at temperature on tensile strength and yield strength is presented in straight-forward fashion as a function of time at each temperature. The strengths shown are for tension tests carried out at the temperature of exposure. The fatigue data likewise are presented in the usual form of stress *versus* number of cycles to failure. Two kinds of creep data are shown: the stress for various amounts of total extension up to and including rupture and the stress for various rates of extension. A logarithmic scale has been used throughout for time, cycles of stressing, and rate.

The extension in creep is the total extension which has taken place from the condition of zero load—in other words, both the elastic and plastic extension during loading, as well as the subsequent creep. It is possible to estimate the amount of creep from the data presented by comparison of the stress in the test to the yield stress at the temperature of test. For loads well below the yield stress, the initial extension can be considered to be elastic only and can be calculated approximately by using the standard value of the modulus of elasticity at room temperature, for example, 6.5 million psi. For stresses in the neighborhood of the yield strength, an additional 0.2 per cent should be added since the yield strength is defined as the stress at which the stress-strain curve deviates by 0.2 per cent from the modulus line. The resultant loading extension is then subtracted from the total extension to give the creep extension. For design purposes, it was felt that the value of total extension was more valuable than creep extension only.

The rates shown apply to the second stage or steady state of creep, which can be observed on most creep curves. Its usefulness in design is considered to be considerably less than that of total extension values. However, it has been widely used in the past for some material and so is given in this paper for the sake of completeness.

TABLE II.—SPECIAL DESIGNATIONS.

○	AZ63	ø ø ø ø ø	1. Notched Fatigue 2. Minimum Creep Rate 3. Creep to Rupture (When Necessary to Distinguish from Other Points)
◓	EM61		
◒	AZ80X		
◐	MI		
◑	EM51		
		○ ○ ○ ○ ○	Used on 0.5% Total Ext. Points to Seperate 0.2, 0.5 and 1.0% Total Ext. Points Lying in a Group.

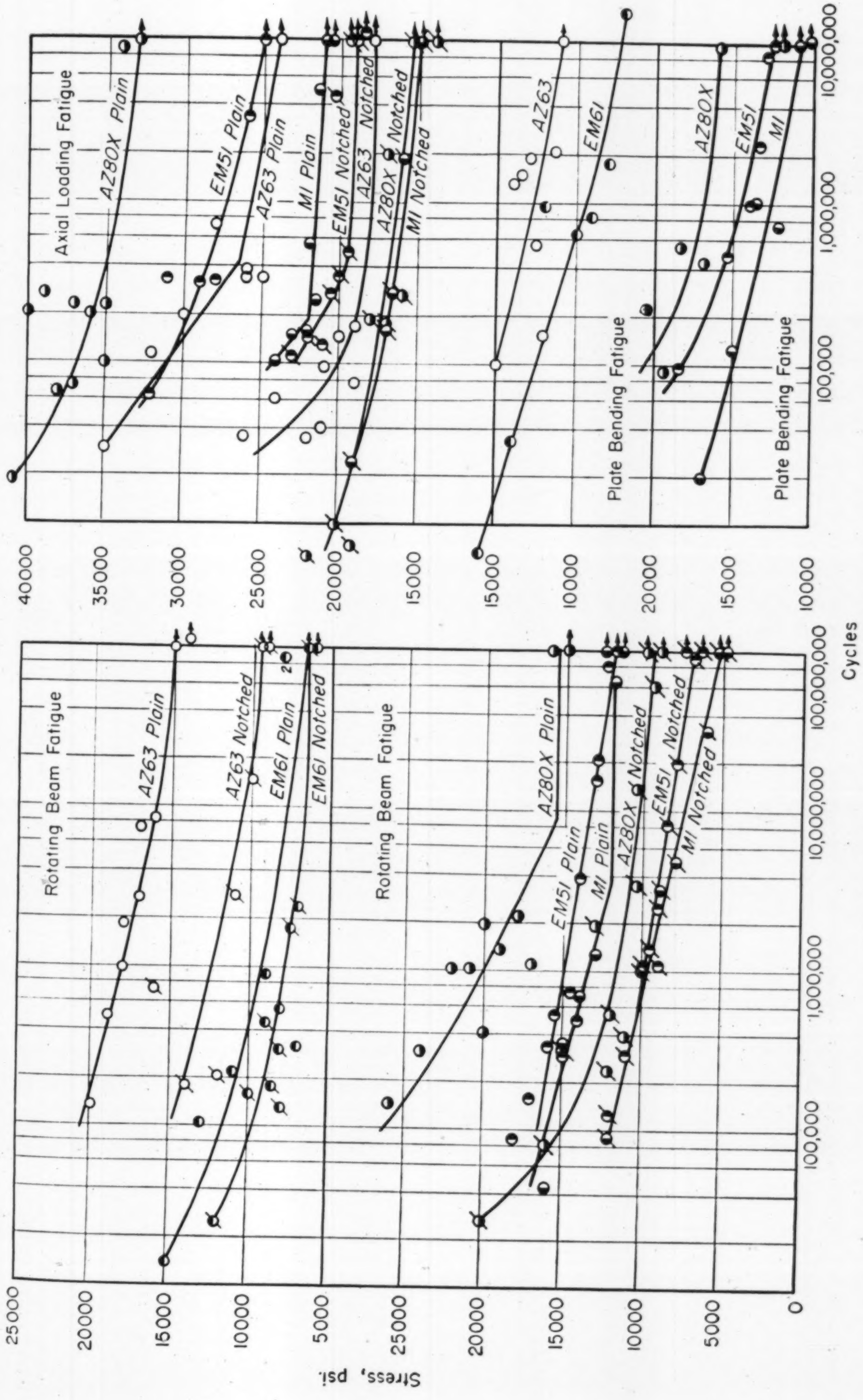


FIG. 1.—Fatigue Strength at Room Temperature.

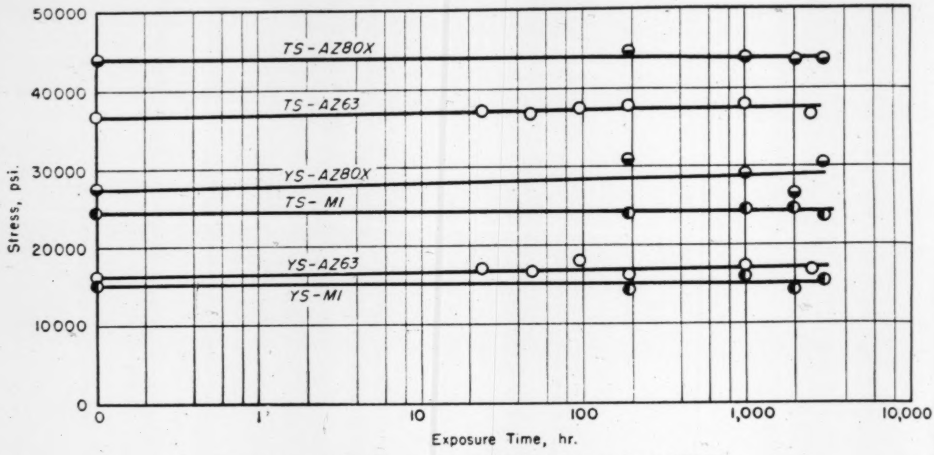


FIG. 2.—Tensile Strength and Yield Strength at 200 F.

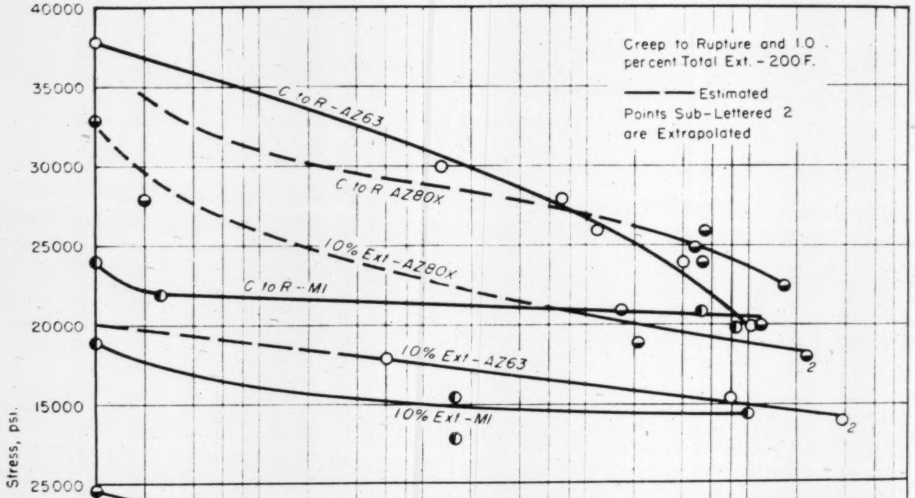


FIG. 3.—Stress for Rupture, and for 1 Per Cent, 0.5 Per Cent and 0.2 Per Cent Total Extension; and Minimum Creep Rate at 200 F.

There are considerable variations in the test results judged by the amount of scatter in the data. Since all test points are given, the reader may judge for himself how much weight to put on any particular curve. The causes of the scatter have not been studied in detail but are probably a result of variations in the original stock as well as variations in testing technique. In certain cases, values have been estimated by interpolation or extrapolation. Such points are indicated on the graph. Estimated curves are shown dotted; such estimations are considered to be of some value and can serve until actual test data are obtained.

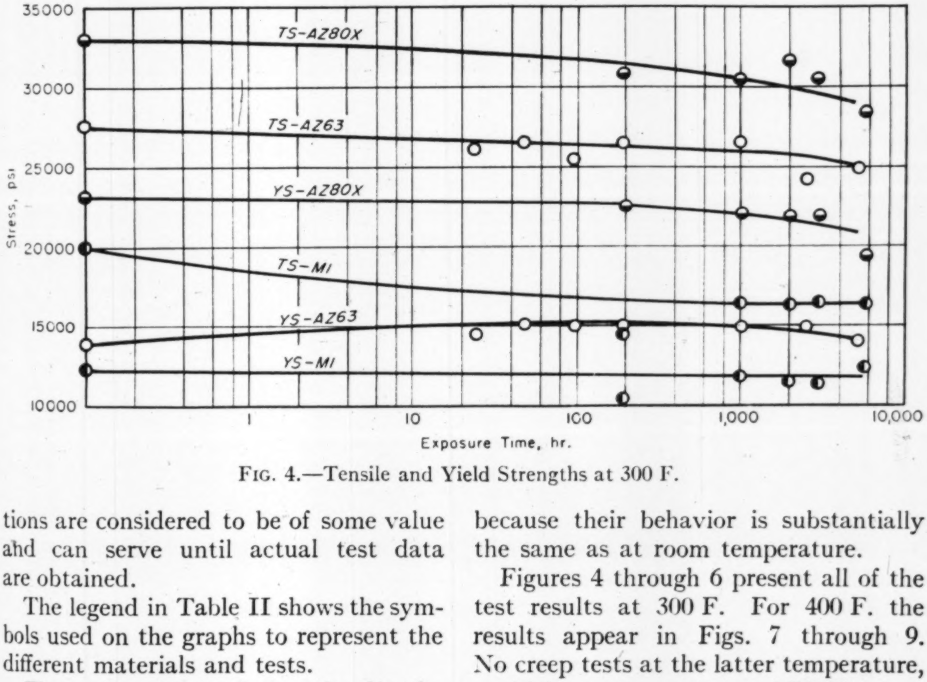


FIG. 4.—Tensile and Yield Strengths at 300 F.

The legend in Table II shows the symbols used on the graphs to represent the different materials and tests.

Figure 1 presents the results for the fatigue tests at room temperature. This presentation was considered to be necessary in order to afford the proper comparison to the results at elevated temperatures. The scatter of the test points is representative of the usual results of fatigue tests obtained in this laboratory on magnesium alloys. This is probably a consequence in part of the rather flat type of curve which is usually obtained on magnesium-base alloys. A small change in stress represents a large change in number of cycles to failure. The test bars for the plate bending tests on EM61 were obtained by machining from a large commercial type casting. No grain refining treatment was used on this melt.

Available data at 200 F. are presented in Figs. 2 and 3. No fatigue tests were run. No tests were made at this temperature on cerium-containing alloys because their behavior is substantially the same as at room temperature.

Figures 4 through 6 present all of the test results at 300 F. For 400 F. the results appear in Figs. 7 through 9. No creep tests at the latter temperature, or higher, were run on AZ80X, since previous work had indicated that it was quite plastic at this temperature. Fatigue tests were run, however, and may be applicable for certain types of service in which the loading is completely reversed so that creep is not critical.

At 500 F. and 600 F., testing was confined to the cerium-containing alloys.

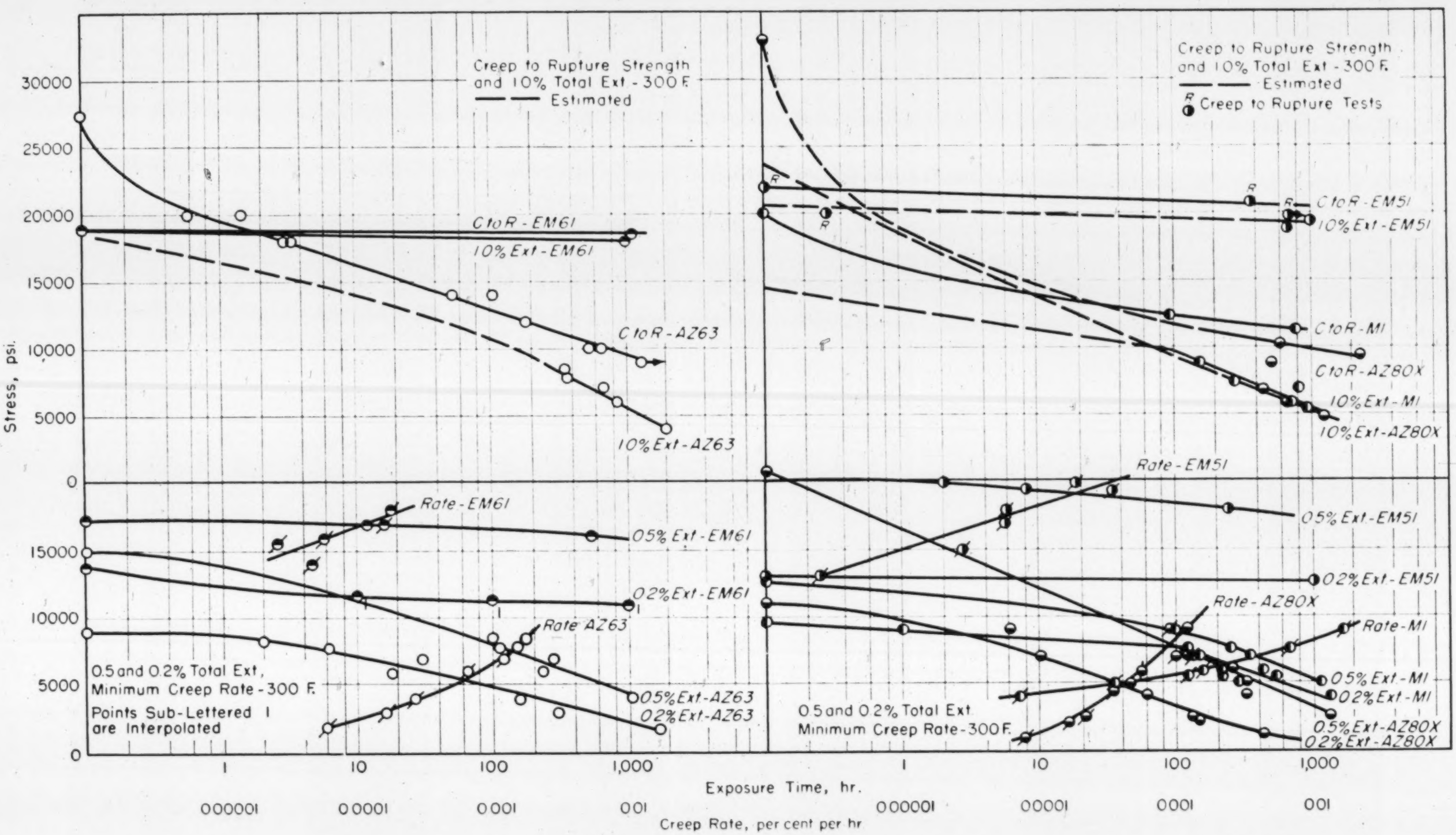


FIG. 5.—Stress for Rupture, 1 Per Cent, 0.5 Per Cent, 0.2 Per Cent Total Extension, and Minimum Creep Rate at 300 F.

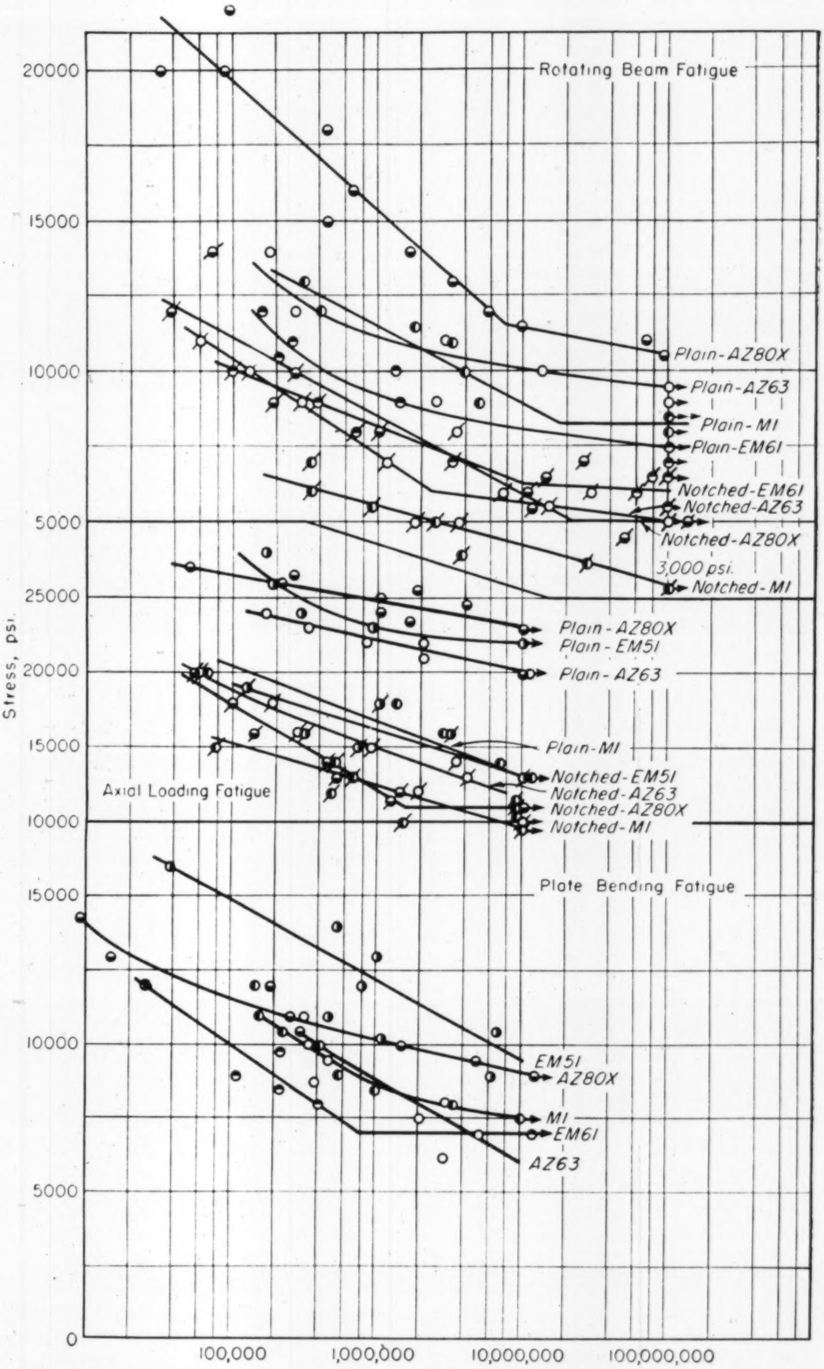


FIG. 6.—Fatigue Strength at 300 F.

Previous work had shown that at these temperatures AZ63 and M1 behaved like AZ80X at 400 F. Figures 10 through 14 present the available test data at 500 F. and 600 F. No fatigue tests were run at 500 F. The plate bending fatigue data on EM61 at 600 F. was also obtained by cutting out test bars from the same commercial casting mentioned previously. Low points, not used in drawing the curves, were found to be associated with specimens of excessively large grain size.

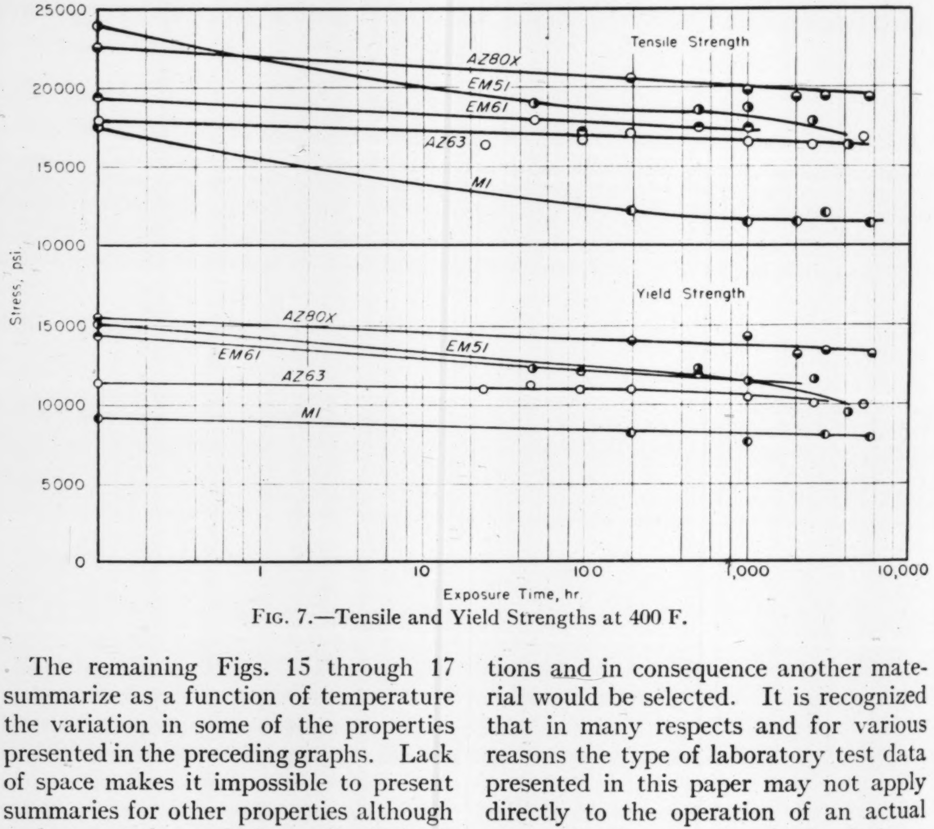


FIG. 7.—Tensile and Yield Strengths at 400 F.

The remaining Figs. 15 through 17 summarize as a function of temperature the variation in some of the properties presented in the preceding graphs. Lack of space makes it impossible to present summaries for other properties although such summaries could be easily derived.

DISCUSSION

The primary use to which these data should be put is the selection of a suitable material for a given set of operating conditions. For aircraft engines there are usually two sets of conditions, maximum and normal. For each condition the designer should furnish the expected type of stressing, magnitude of stressing, time of exposure and temperature. Reference to the graphs should then be made to select a material which will meet both maximum and normal operating conditions. In some cases a material might be satisfactory under normal conditions but not under maximum conditions and in consequence another material would be selected. It is recognized that in many respects and for various reasons the type of laboratory test data presented in this paper may not apply directly to the operation of an actual part. On the other hand, the designer may have an equally difficult job presenting estimates of operating conditions which will apply to the operation of the part. At the least, the best estimate of operating conditions used in conjunction

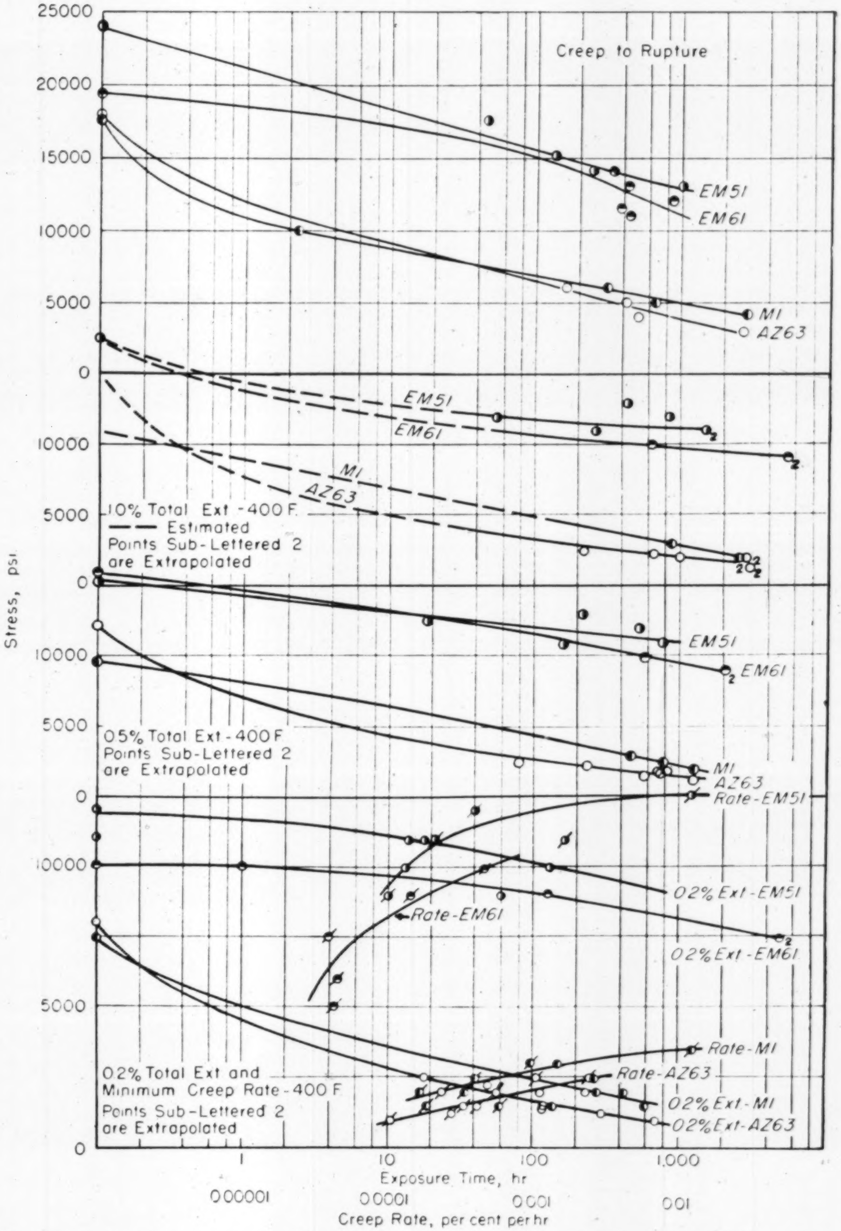


FIG. 8.—Stress for Rupture, 1 Per Cent, 0.5 Per Cent, and 0.2 Per Cent Total Extension, and Minimum Creep Rate, at 400 F.

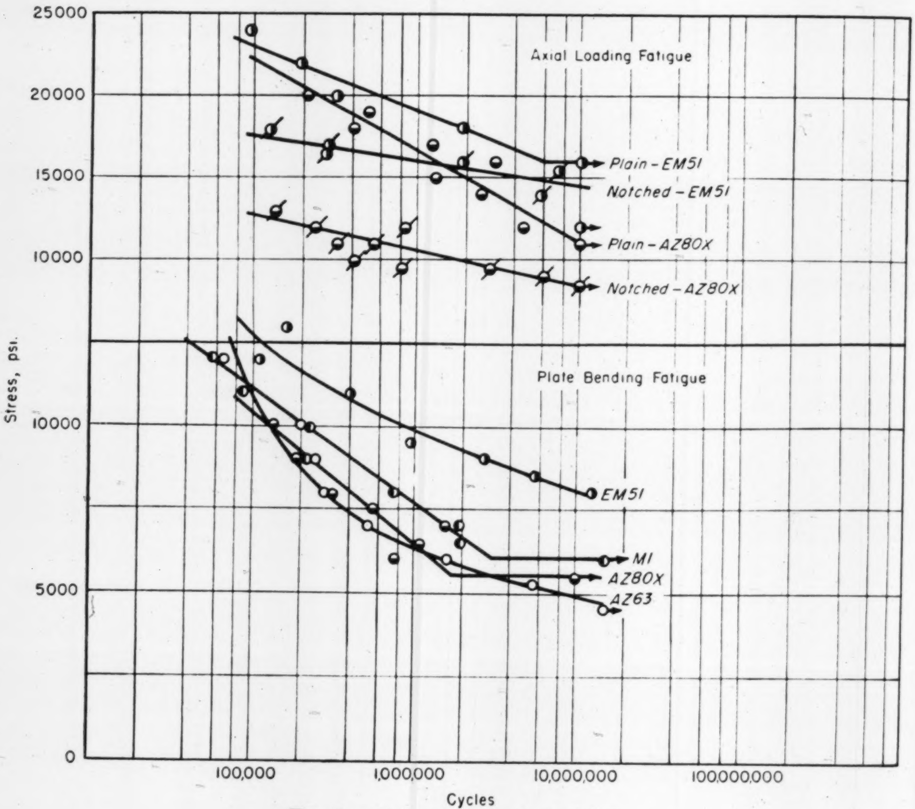


FIG. 9.—Fatigue Strength at 400 F.

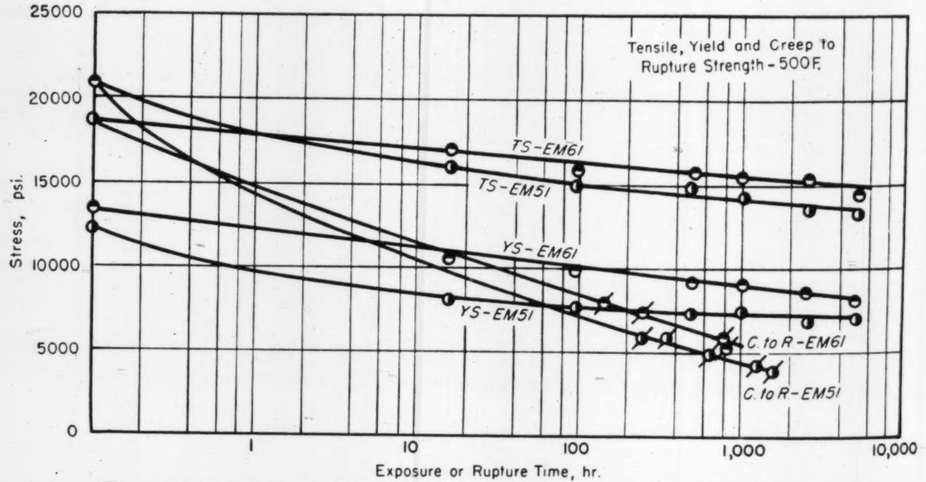


FIG. 10.—Tensile Strength, Yield Strength, and Stress for Rupture at 500 F.

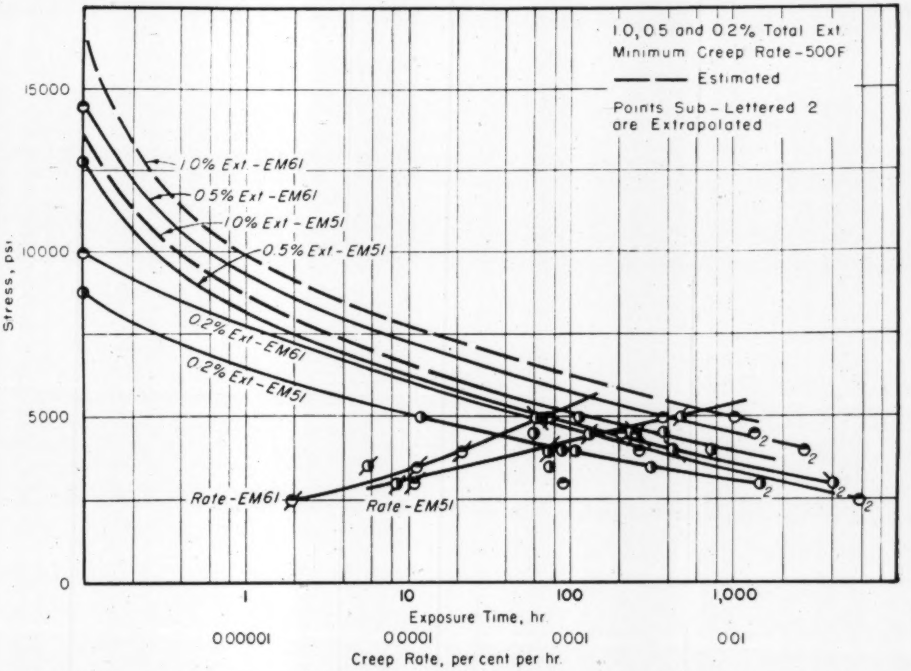


FIG. 11.—Stress for 1 Per Cent, 0.5 Per Cent and 0.2 Per Cent Total Extension; and Minimum Creep Rate at 500 F.

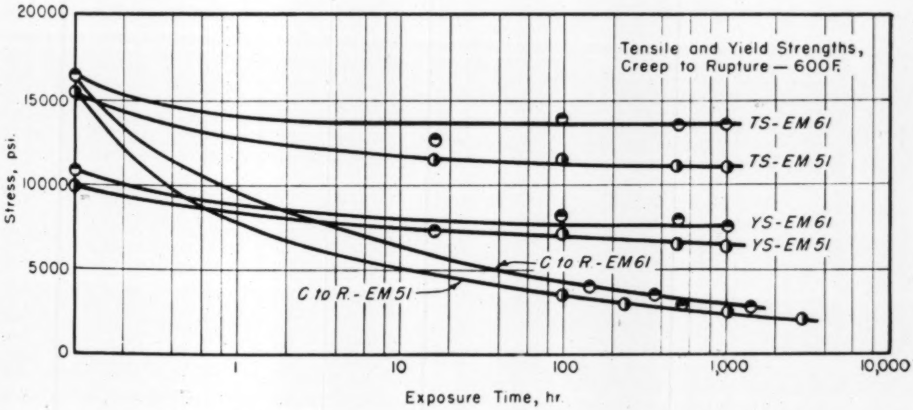


FIG. 12.—Tensile Strength, Yield Strength and Stress for Rupture at 600 F.

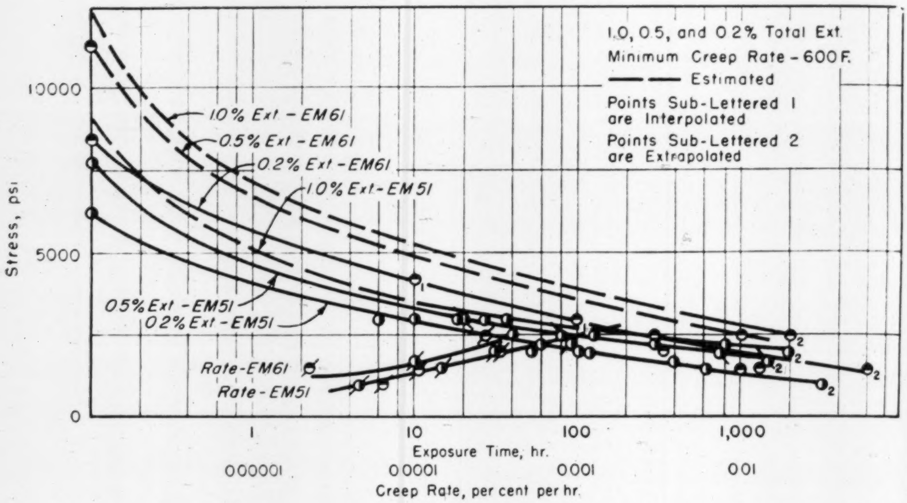


FIG. 13.—Stress for 1 Per Cent, 0.5 Per Cent and 0.2 Per Cent Total Extension, and Minimum Creep Rate at 600 F.

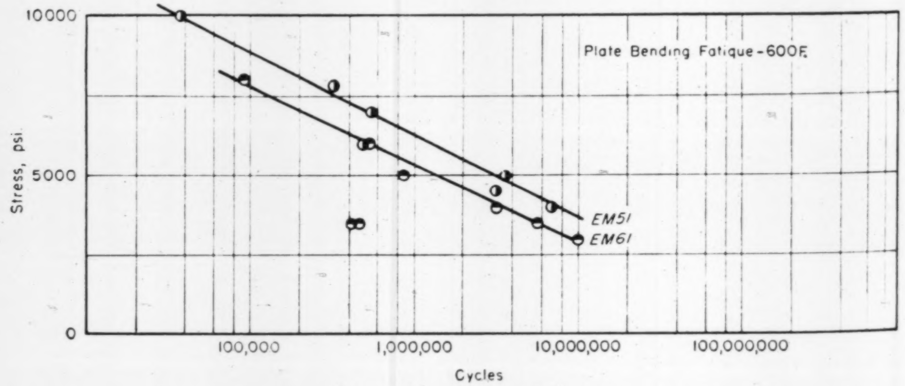


FIG. 14.—Fatigue Strength at 600 F.

with the data presented in this paper will furnish the most intelligent selection of a material that it is possible to make today.

The graphs may also be used to make some very general comparisons of the merits of the respective alloys tested. This procedure has its dangers since any statements made can only be generalities and cannot take the place of the procedure described in the preceding paragraph for the proper selection of a material. Within these limits certain statements can be made, based particularly on the summary results of Figs. 15 through 17. In Fig. 15 the generally steeper slope of the curves for the aluminum or manganese-containing alloys up to 400 F. is indicative of the superiority under tensile loading beyond this temperature of the cerium-contain-

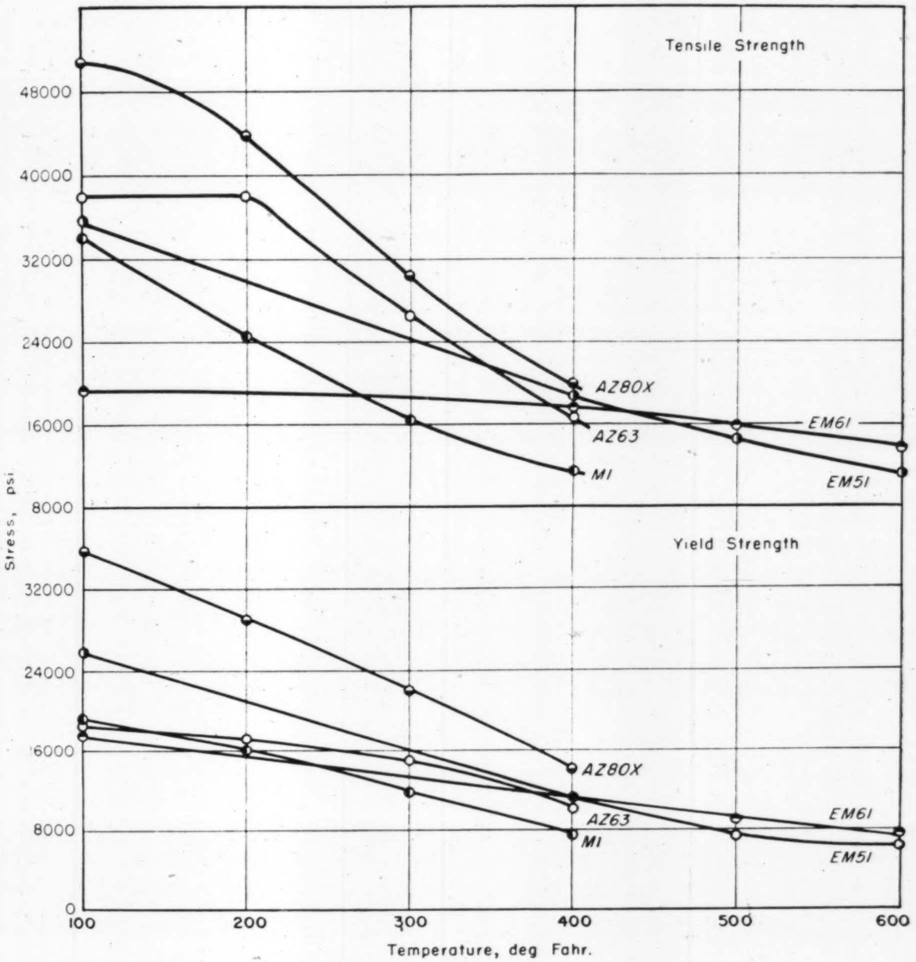


FIG. 15.—Tensile Strength and Yield Strength at Temperature after 1000 hr. Exposure at that Temperature.

ing material. The effect is also shown by the yield strengths given in Fig. 15. However, the creep curves plotted in Fig. 16 show much more clearly the extent to which the cerium-containing alloys are superior to the aluminum or manganese type. The superiority is quite evident above 300 F. These two figures also show that under some conditions the manganese-containing alloy, M1, is better than the aluminum-containing alloy, AZ80X. For fatigue loading, however, the AZ80X appears to be better than the M1 as seen in Fig. 17. All of the above effects can be seen more clearly by reference to Figs. 1 through 14, which present the data in much greater detail than can be shown in the summarizing Figs. 15 through 17.

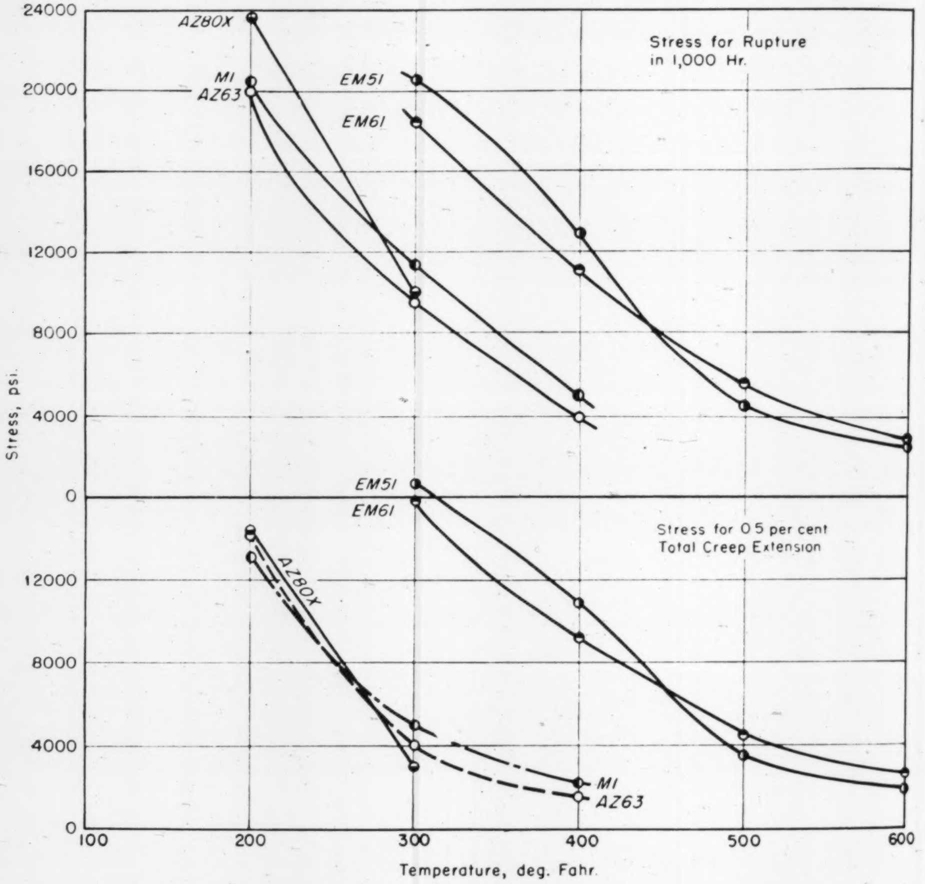


Fig. 16.—Stress for Rupture and for 0.5 Per Cent Total Extension in 1000 hr. for the Various Test Temperatures.

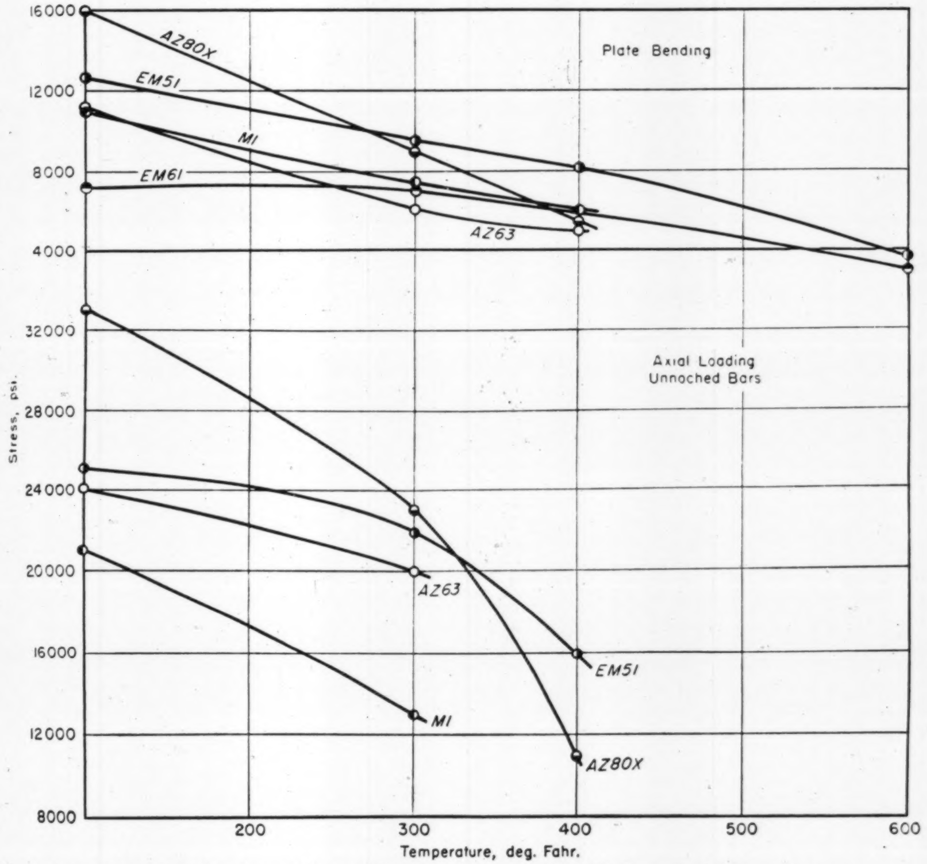


FIG. 17.—Fatigue Strength at 10,000,000 Cycles for the Various Test Temperatures.

REFERENCES

(1) A. A. Moore and J. C. McDonald, "Tensile and Creep Strengths of Some Magnesium-Base Alloys at Elevated Temperature," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 970 (1946).

(2) G. H. Found, "The Notch Sensitivity in Fatigue Loading of Some Magnesium-Base and Aluminum-Base Alloys," *Proceedings*, Am. Soc. Testing Mats. Vol. 46, p. 715 (1946).

(3) T. E. Leontis and J. P. Murphy, "Properties of Cerium-Containing Magnesium Alloys at Room and Elevated Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 166, p. 295 (1946).

(4) T. E. Leontis, "The Room and Elevated Temperature Properties of Some Sand Cast Magnesium-Base Alloys Containing Zinc," *Metals Technology*, Am. Inst. Mining and Metallurgical Engrs., June, 1948, p. TP2371.

DISCUSSION

Mr. Jay R. Burns[1] *(by letter)*.—The test data in this paper are certainly of interest to the user of magnesium alloys for engine parts. The purpose for which the data are intended may be furthered by the following test results obtained at the Materials Laboratory, Wright-Patterson Air Force Base. All samples for testing were machined from a 35-lb. E6 alloy cast and HTA compressor casing. The room temperature tension tests represent a survey of the casting to determine its maximum and minimum room temperature properties. Elevated temperature tension tests were made in accordance with A.S.T.M. procedures and a crosshead speed of 0.1 in. per minute.

Test Temperature, deg. Fahr.	Tensile Strength, psi.	Elongation in 1 in., per cent
Room (14 Bars)	Average 12 600	1.0
	Maximum 14 000	1.0
	Minimum 11 100	1.0
400 (2 Bars)	13 200	4.5
600 (4 Bars)	9 400	42

The stress rupture values obtained on ½ by 2-in. gage length bars are as follows:

Test Temperature, deg. Fahr.	100-hr. Stress Rupture, psi.	1000-hr. Stress Rupture, psi.
400	13 500	11 000
600	3 200	2 250

The 100-hr. stress rupture figure is 300 psi. greater than the short-time tensile strength. This is undoubtedly due to the combination of a variation of properties through the casting and a small rate of decrease of stress rupture values with time. A coarse grain size in the casting (grain diameters of from 0.020 to 0.060 in.) is believed to be largely responsible for the difference in properties of test bars cut from this casting and properties of separately cast test bars.

All bars tested at 400 F. were stabilized 72 hr. at this temperature prior to testing and likewise bars tested at 600 F. were stabilized 48 hr. at 600 F.

---

[1] Air Materiel Command, Dayton, Ohio.

SOME CHARACTERISTICS OF RESIDUAL STRESS FIELDS DURING DYNAMIC STRESSING ABOVE THE ENDURANCE LIMIT*†

By James B. Duke[1]

Synopsis

Samples of S.A.E. 1020 and S.A.E. 4130 steel were investigated during bending fatigue tests using a comparative magnetic process to determine both fatigue damage and the direction and relative magnitude of residual stress fields set up during the endurance life of the metal.

Results indicate that valuable information on the progress of plastic deformation due to differential cold working, prediction of failure from fatigue, and several magnetic properties not easily measured can be obtained by a simple testing technique regardless of heat treatment or composition of the steel.

Recent developments in the field of electronic research, particularly when applied to magnetic analysis of metals have led several investigators to a reexamination of the possibilities of determining basic properties of steels by comparative magnetic methods (**1**, **2**).[2] A series of experiments utilizing the Cyclograph have yielded encouraging results (**3**, **4**).

The theoretical basis for such investigations has been determined by several authors, but the bridge between theoretical relationships and practical experimental techniques have been slow in developing and are, as yet, lacking in both volume and accuracy for definite conclusions.

This paper considers a very important aspect of material strength in investigating residual stresses set up during dynamic loading. Any information which can be obtained on these stresses, even if only comparative, will serve the engineer well, and if the technique yields the possibility of nondestructive tests for dynamic failure, its further exploration should be assured.

Magnetic Factors

One important retardent to any investigation of a ferromagnetic metal for its basic properties is the very complexity of the phenomena involved. It is often impossible to isolate the interaction of magnetic effects.

The DuMont Cyclograph used in these experiments measures, on a relative basis, the hysteresis and eddy current losses set up by the insertion of a metallic core in a high-frequency coil which is supplied with current from a highly sensitive oscillator. The influence of changes in the crystal structure of the core due to stresses has the effect of changing the coil field intensity and the output of the oscillator. This signal is transferred through an electronic circuit to a cathode ray tube from which measurements may be made. However, it is impossible to prevent certain characteristics of the instrument from imposing themselves on the readings, so that quantitative results are impractical. Determination of the base or zero for magnetic losses is a very difficult procedure and can be accomplished only in certain isolated cases. Separation of eddy current and hysteresis losses in a ferromagnetic material has not been wholly successful.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

† Experimental work was carried out in the laboratories of Massachusetts Institute of Technology under the guidance of the faculty and staff.

[1] Vibrations Department, Hamilton Standard Propellors Div., United Aircraft Corp., East Hartford, Conn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 762.

A distortion of the crystal lattice in a sample, whether induced by static or dynamic stressing, will have an effect on the magnetic structure of the metal. Statically, it is well known that, with an increase in stress, the hysteresis losses and the specific resistivity of a metal increase. These increases are accompanied by corresponding changes in the coercive force, permeability, and remanence of material of positive magnetostriction.

The foregoing statements are true if measurements are made while the material is stressed, that is, an increase in stress results in an increase in the hysteresis loss and the specific resistivity. In this investigation, the same is assumed true for residual stresses set up by a reversed-bending fatigue test. These residual stresses are isolated from the stress induced by the dynamic loading by periodic investigation of the material when entirely free of external load.

An exact relationship for the form of the hysteresis (H), stress (S) curve has not been determined, but it can be approximated roughly by an exponential equation of the form:

$$H = KS^a$$

where:

H = the net magnetic loss due to hysteresis effects alone,

K = a constant depending on the material permeability, coercive force, and remanence,

S = the internal stress, and

a = a constant whose value depends largely on the internal stress condition prior to testing.

The foregoing relationship is only for purposes of simplification, but it will be true regardless of the initial magnetostrictive state of the material within the stress limits of these experiments.

The electromotive force causing eddy current losses in the sample is constant for a particular test coil frequency so that an increase in resistivity with an increase in internal stress will cause a corresponding decrease in the eddy current loss following the relation:

$$W = \frac{AE^2}{R}$$

where:

W = the losses due to eddy current losses alone,

A = a constant depending on the hardness, surface condition, and size of the material,

E = the electromotive force, and

R = the specific resistivity of the material.

Initially, the hysteresis losses far overshadow those from eddy currents, but inasmuch as measurements are made on a relative basis to these initial losses, the behavior of the Cyclograph may be controlled by either. The instrument will measure net loss differences during the test process.

It would be expected that, operating at stresses at or above the endurance limit, the material would undergo extensive lattice destruction which would be accompanied by severe changes in the magnetic domain structure. From theoretical relationships, a reversal in the magnetostriction is expected which will result in a decrease in resistivity with a further increase in internal stress (5). This will cause a corresponding increase in the eddy current losses making them additive to the already increasing hysteresis effects. This Villari reversal in the magnetostriction is not an easy phenomenon to measure, but it is readily apparent in the experimental results obtained in this paper.

SUMMARY OF TESTING PROCEDURE AND EXPERIMENTAL RESULTS

Specimens were prepared of S.A.E. 1020 steel in the fully annealed, normalized, and as rolled conditions, and of S.A.E. 4130 steel which had been quenched and drawn, and normalized. The samples were in the form of flat strips $\frac{1}{8}$ by 1 in., with a length adjusted to give a desirable resonant frequency in the range of a Rayflex type fatigue tester.

A sensitivity was chosen for the Cyclograph, and the instrument calibrated according to operating instructions. Two samples from each heat-treated condition of the two steels were chosen as "standard samples" upon which to base Cyclograph settings. The pieces were stressed dynamically at 120 cycles per second in reversed banding at stresses above and below the endurance limits (6).

The samples were stressed for a definite period, 25,000 cycles, and then removed from the fatigue tester and examined in the Cyclograph with test frequency coils of 4.5, 5.0, and 9.0 kilocycles. Prior to examination, each sample was thoroughly demagnetized using standard procedure. Utilization of the standard samples insured accurate setting of the Cyclograph at the same values which had existed at the beginning of the test.

A constant stress was maintained during the entire dynamic testing process by holding the amplitude of vibration constant. The experimental resonance vibrator is shown in Fig. 1 with associated apparatus. Each test piece was carried through to failure in one working period to prevent extraneous recovery due to lapsed time. Specimens were machined and polished to a consistent surface index of 40 microinches, simulating conditions on major service parts. In the case of S.A.E. 4130 steel, two surface conditions were examined, that of 70 microinches and of 12 microinches, simulating conditions in which that metal most commonly occurs, namely, rough machining and close tolerance pieces.

The specimen was deemed to have

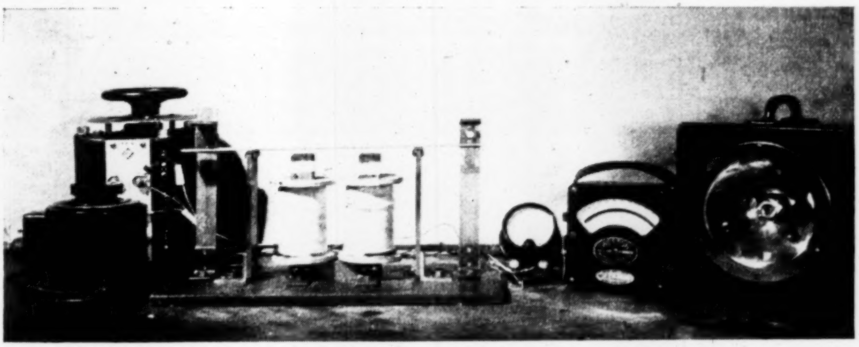


FIG. 1—Magnetic Vibrator and Associated Testing Apparatus.

failed when surface cracks were detected by means of a Zyglo examination and the tests terminated then. In many cases, this initial cracking occurred during a fatigue period, but a constant amplitude was maintained by an increase in power input and the period completed for a final reading from the Cyclograph.

Figure 2 shows typical results for S.A.E. 1020 steel in an as-rolled condition at stresses above and below the endurance limit. It can be seen that the curves for all samples are similar in form regardless of stress and that the magnitude of the relative core losses and the location of points *A*, *B*, and *C* are the determining factors in the failure or nonfailure of the specimen. These points will be discussed in a later section.

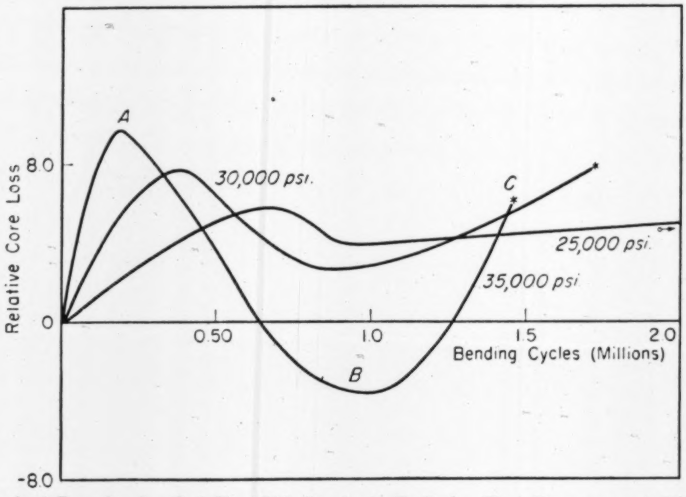


FIG. 2—Typical Results for S.A.E. 1020 Steel, as Rolled. Test Frequency 4 Kilocycles.

Samples of this steel which had been normalized or annealed behaved in a radically different manner. In the case of normalized specimens, the relative losses were constantly negative and decreasing up to about 300,000 reversals when the curve exhibited the characteristic reversal and began to rise slowly to the horizontal axis. The prediction of a fatigue failure was based on the slope of the curve after the reversal, the specimens stressed below the endurance limit having the smaller slope.

Annealed specimens were consistent at all stresses, but the curves had their reversal points in a mirror image of those shown in Fig. 2. The relative losses first became negative, reversed at 100,000 cycles, then became increasingly positive until a second reversal occurred with a corresponding increase in negative losses.

In each case, calibration points established by initial samples served accurately to predict the failure or nonfailure of ensuing test specimens. The effect of an increase in stress served to increase the magnitude of the losses as would be expected from theoretical considerations. The use of different test frequencies allowed examination of definite sections of the material, because effective flux penetration will vary with frequency, thereby enabling isolation of effects into definite layers of the cross-section. The nine kilocycle tests yielded curves similar to those for the lower frequency coils except that the magnitude of the losses was smaller, and once the maximum effects had been attained on the surface layers, the curves leveled off, indicating that no further damage was occurring in these areas.

A typical graph for S.A.E. 4130 steel, quenched and drawn, is presented in Fig. 3 with typical samples stressed below and above the endurance limit in both surface conditions mentioned previously. Those samples with a rough machined surface failed at all stresses examined due to surface stress raisers which aggravated existing fatigue damage. Each curve, regardless of surface index, has similarity to the others at points *D*, *E*, and *F*, and exhibits the same characteristics of slopes, reversals, and prediction of failure from a calibration sample as did the low-carbon steel.

Normalized samples of this material had much greater relative losses and sharper reversal points. The curves rose sharply during the first hundred thousand reversals, reversed and dropped quickly to values slightly above the basic reference loss, and then increased in value during the remainder of the test. As in the normalized 1020 steel, the prediction of failure could be accurately based on the slope of the latter portion of the curve.

Considerable scatter existed in the test points for all samples tested. This was manifested in the high bending cycle portion of the curve as an oscillation of the points above and below the average curves shown in Figs. 2 and 3. None of the tests can be considered as true endurance determinations because of the high surface roughness and uncontrolled heat treatment, but this was not the original purpose of the experiments.

In testing samples of steel at the same stress, considerable variation occurred in the magnitude of the relative core losses, making it difficult to determine the test stress by losses alone, but it was found that the curves were very similar and that, for a particular heat treatment, the reversals in slope occurred at the same points in the bending cycle axis, making the fatigue life predictions mentioned previously accurate within the usual limits of endurance testing.

The results for S.A.E. 1020 steel are

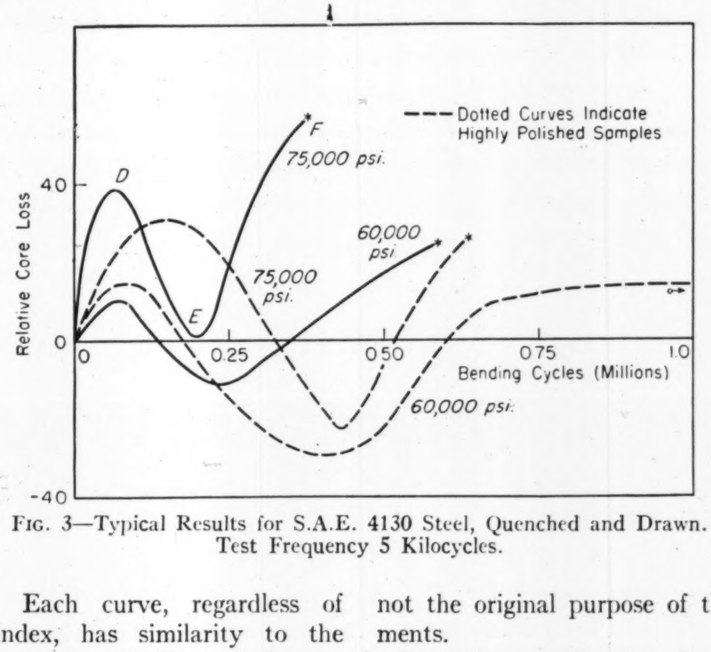


FIG. 3—Typical Results for S.A.E. 4130 Steel, Quenched and Drawn. Test Frequency 5 Kilocycles.

general for low plain carbon steels and will apply to similar steels whose magnetic changes are large whether induced by bending or direct stresses. S.A.E. 4130 steels were tested to show specifically that a magnetic method will be successful regardless of alloying content provided that precautions are taken against errors due to the greater retentivity of the metal.

DISCUSSION OF EXPERIMENTAL DATA

In the interest of brevity, only those samples depicted in Figs. 2 and 3 will be discussed in this section. Although the various heat treatments of the two steels behaved in entirely different manners from those to be discussed, a theoretical analysis can be made which is logical and consistent with that to follow. Verification of the analysis has been partially accomplished by other investigators (2, 3, 4) and in previous work of this author (7).

Two types of internal stresses are to be considered in any fatigue test for accurate analysis. The first of these, the macroscopic stresses, are induced by the type of loading and are locked within the material in equilibrium after the loads are removed. The type of stress distribution is largely dependent on the second type of stresses, the so-called microscopic stresses which are randomly distributed within the individual crystals and are induced by cold work, intensity of heat treatment, magnetostrictive strain, and by the macroscopic stress. This interdependence between the two types makes more difficult the isolation of effects due to one or the other. In general, the magnetic effects will be most largely affected by the macroscopic stresses, but these, in turn, are considerably altered by the microscopic fields. A well-known treatise on ferromagnetism yields the two basic curves on which this analysis is based (8).

An initial positive magnetostriction is assumed for both types of steel investigated.

Samples of S.A.E. 1020 steel, cold rolled, are under an initial core compression and surface tension as the tests begin, due to the longitudinal rolling process. The dynamic loading relieves the core stresses in the first hundred thousand reversals between points *O* and *A*, Fig. 2. This relief is elastic in nature and accomplishes a state of magnetic neutrality by domain boundary movement into the lines of applied stress. This effect serves to establish a new magnetic reference level at point *A* because of the greater ease of magnetization, and to increase the net core loss. Between points *A* and *B*, the core loss curves drop rapidly as the number of fatigue cycles increase. This is due to a semi-elastic, semi-plastic deformation which is progressive in severity. By this statement, we mean that the progress of plastic deformation is by layers from the outer surface inward and that plastic flow on an inner layer begins only upon complete flow of the adjacent outer section. In this region the decrease in the core loss due to increasing resistivity is the predominate factor. The effective field of the test coils penetrates farther into the sample as the material undergoes differential flow. Domain boundary movement is replaced by the beginnings of boundary rotation as the curve approaches point *B*. Increased macroscopic stress and intensified microscopic fields tend to induce a mixed state of positive and negative magnetostriction which reduces the effects of increasing resistivity to give a large curvature to the lower portion of the curve in the region of *B*.

At *B*, the magnetostrictive state has become almost wholly negative indicating that the material has experienced the Villari reversal. This reversal can be associated with the completion of the differential plastic flow so that the region between *B* and *C* indicates stressing entirely in the plastic region. The reversal in the magnetostriction results in decreasing resistivity as the stresses build up and the hysteresis and eddy current losses are, for the first time, additive. The energy which has produced plastic flow in the sample is now directed to lattice destruction, the intensity of which is entirely dependent on the applied stress once the sample has reached point *B*. The extent of this lattice destruction, which is associated with rapid progression of the fatigue failure to the formation of incipient cracks, affects sharply the slope of the core loss-bending cycle curve and enables prediction that a failure will occur, usually within a hundred thousand cycles.

From consideration of magnetic domain theory, it appears that the major residual stress effect is due to a predominance of compressive fields which induce an early flow and rapid destruction of the crystal bonds. Samples which did not fail produced core loss-bending cycle curves similar to samples which did fail up to point *B*. After this point, the slope of the curve was much less and leveled off at a core loss value slightly above the reference axis at the beginning of the test.

The oscillation of the test points about the average curve shown in Fig. 2 emphasizes the differential cold working mentioned above. At each transition it would be expected that the increased depth of field penetration would yield a greater loss, but in this region, increasing resistivity is the predominate factor and the net effect drops the test point below the average curve. If the subsequent loading period is not sufficient to stress this layer to high flow, the following point will evidence a lesser resistivity effect, and falls above the average curve.

It should be noted that lower stresses delay the characteristic reversals along the bending cycle axis. This makes it possible to approximate the fatigue stress and serves as an accurate indication of the portion of the fatigue life that the specimen lies in at any time.

Samples of S.A.E. 4130 steel, quenched from 1650 F. and drawn at 1100 F., exhibited similar characteristics to the lower carbon steel. However, since this material is more highly internally stressed and has much higher sensitivity to domain activity, the losses were expected to oscillate more radically. For this reason the Cyclograph sensitivity was adjusted to one third that of the previous tests.

The analysis is similar to that for the lower carbon steel. The initial loading period serves to align the domains into the lines of stress, thereby raising the magnetic level. Following this, a period of rapid differential cold working occurs until the material is in a fully plastic state at point *E*, which is associated with the Villari reversal and the beginning of active fatigue destruction. The rapidity of this destruction at high stresses makes the curve rise sharply in samples which are destined to fail and in a more gentle manner for those specimens stressed below the endurance limit.

The only apparent difference between the two surface indexes tested is a more gradual development of the characteristic curves which allows the predominate effect of increasing resistivity in the region *D* to *E* to pass the relative loss below the initial zero setting. Polished samples are tested at the same stresses as the rough index specimens so that curves may be compared. The polishing enabled testing of specimens through two million cycles at a stress slightly below the endurance limit. Failure occurred in all the high index specimens in a manner similar to tests performed by other investigators (9).

A high internal microscopic stress concentration serves to accelerate all phases of the test, particularly from E to F, the failure portion. This is not an unusual conclusion, but this test graphically illustrates the effect. Similarly, the polished samples serve to show that, no matter what the initial stress conditions are, the macroscopic stresses will intensify the fields in proportion to the aggravation present on the surface and interior of the sample.

The ease and facility of the testing procedure, and the fact that Cyclograph coils can be made to reach a great many service parts, which can be tested in place while not under load, make desirable a further exploration of magnetic tracing techniques. The results given in this paper are based on a limited number of experiments, but there is every indication that good agreement exists between theory and test, and that valuable information may be obtained by calibration of test samples and application to materials of the same composition undergoing service stressing.

Acknowledgments:

The author would like to express his appreciation to Mr. P. E. Cavanagh of Cyclograph Services Ltd. for providing the equipment necessary for these experiments, and for constant encouragement and advice during the actual work, and to Mr. T. W. Wlodek of the Canadian Department of Mines and Resources for his close cooperation and verification of the test results.

REFERENCES

(1) T. W. Wlodek, "A Magnetic Analysis of the Progress of Plastic Deformation During Dynamic Loading," Thesis, Massachusetts Institute of Technology (1947).

(2) P. E. Cavanagh, "Stress Comparisons by Correlation with High Frequency Magnetic and Eddy Current Losses," *Transactions*, Am. Soc. Metals, Vol. 36, p. 137 (1946).

(3) P. E. Cavanagh, "Some Changes in the Physical Properties of Steels and Wire Rope During Fatigue Failure," *Bulletin*, Canadian Inst. Mining and Metallurgical Engrs., July, 1947.

(4) P. E. Cavanagh, "The Progress of Failure in Metals as Traced by Changes in Magnetic and Electrical Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 639 (1947).

(5) F. Bitter, "Introduction to Ferromagnetism," McGraw Hill Book Co., Inc., New York, N. Y. (1937).

(6) H. F. Moore and J. B. Kommers, "The Fatigue of Metals," McGraw Hill Book Co., Inc., New York, N. Y. (1927).

(7) J. B. Duke, "A Qualitative Investigation of the Core Losses in Metals with Appoaching Fatigue Failure," Thesis, Rensselaer Polytechnic Institute (1945).

(8) R. Becker and W. Doring, "Ferromagnetismus," Edwards Bros., Inc. (1943).

(9) J. A. Bennett, "A Study of the Damaging Effect of Fatigue Stressing on X4130 Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 693 (1946).

DISCUSSION

Mr. P. E. Cavanagh.[1]—Apart from the possible practical inspection applications of this method as outlined by Mr. Duke, there is another fundamental point which should be emphasized. Internal micro-stresses can be followed magnetically, measured and traced during fatigue stressing. Whether the numerical values of micro-stresses that can be obtained magnetically are the true values which will tell us in advance something of the behavior of metals under different modes of stressing must be determined experimentally. That is an important point about this paper. It is one approach to the problem of determining whether magnetically measured micro-stresses can be used to predict the mechanical behavior of metals.

I should like also to call attention to another paper presented at this Annual Meeting by Mr. Lamson, "The Evaluation of Hysteresis Core Loss by Power Equations."[2] In this paper there was a very excellent and complete explanation of the mechanics of magnetization due to external force, namely, the magnetizing field. The remarks of Mr. Lamson can be applied equally well to the magnetization processes brought about by external stresses.

One fundamental thing brought out in Mr. Lamson's paper is that the "magnetic elastic limit" is of the order of two oersteds. Any work similar to that done by Duke must be carried out at a field strength below this level, for the same reasons that damping capacity is measured below the mechanical elastic limit.

Mr. Burt L. Newkirk[3] (*presented in written form*).—Mr. Duke has made a notable contribution toward the problem of determining fatigue damage by nondestructive test. A number of students have studied phases of this problem over a period of several years at Rensselaer Polytechnic Institute, using apparatus similar to that shown in the accompanying Fig. 4 to fatigue the specimens. One group studied the change in natural frequency of the specimen and in the internal friction of the metal as fatigue progressed.[4] These properties showed trends but very little change as fatigue progressed.

In the course of these tests it has been noted that considerable changes occurred in the power required to vibrate the specimens at the amplitude necessary to maintain the stress range. Where the stress range was such as to cause failure after more than a million reversals, the power requirement usually increased gradually as fatigue progressed. Recently Edward Widmayer, a graduate student, studied this factor using specimens of cold-rolled steel in the as-received and in the annealed condition. The annealed specimen showed a definite trend indicated by the curve in the accompanying Fig. 4. In these tests, the stress range exceeded the yield value and failure resulted after 500,000 to 600,000 reversals.

---

[1] Assistant Director, Dept. of Engineering and Metallurgy, Ontario Research Foundation, Toronto, Ontario, Canada.

[2] Symposium on Magnetic Testing, Am. Soc. Testing Mats. (1948). (Symposium issued as separate publication *STP No. 85*.)

[3] Professor of Vibration Theory and Practice, Rensselaer Polytechnic Institute, Troy, N. Y.

[4] Discussion of paper by E. Epremiam and E. F. Nippes, "The Fatigue Strength of Binary Ferrites," *Transactions*, Am. Soc. Metals, Vol. 40, (1948).

It would seem that efforts should be continued to find additional nondestructive tests for the progress of fatigue damage.

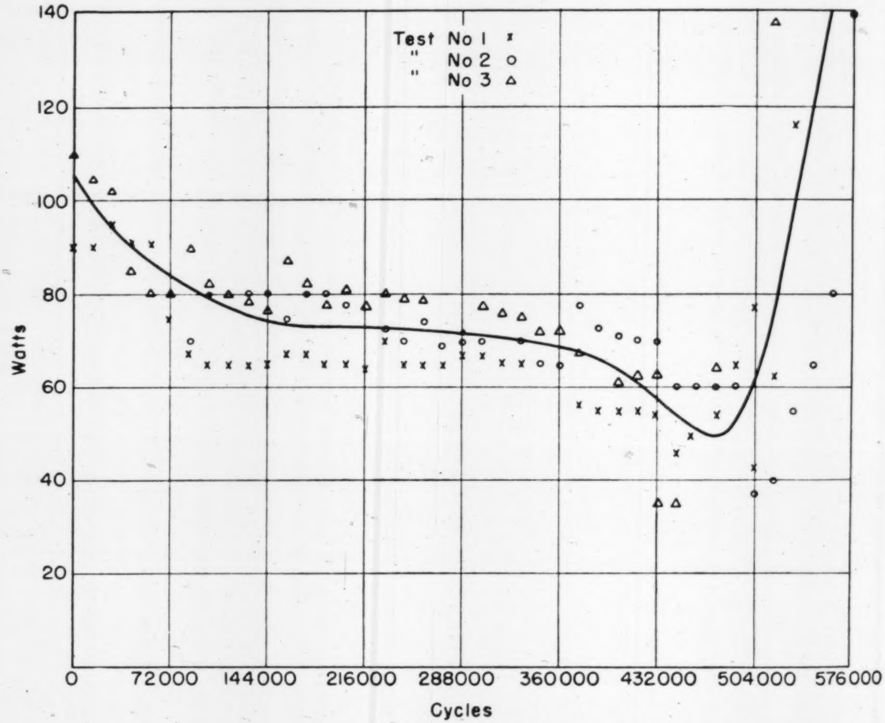


FIG. 4.—Power *versus* Cycles.

Cold-Rolled S.A.E. 1020 steel. Annealed 40 min. at 1600 F. Furnace cooled. Stress, 38,000 psi. Yield Point 34,000 psi.

MR. T. J. DOLAN.[5]—I should like to raise several questions for consideration by the author. In connection with the term "residual stresses measured by the electrical means," I presume from the previous work of Mr. Cavanagh that they are of a micro nature. I am wondering whether the method employing electrical measurements has the ability to differentiate between favorable and unfavorable types of residual stress and plastic deformation as far as fatgue strength is concerned.

Briefly, my questions would be somewhat as follows:

1. Do the electrical measurements differentiate between favorable and unfavorable types of residual stress?

2. Is there any direct correlation or relation between mechanical hysteresis and the electrical core losses or electrical hysteresis as measured by these tests?

3. Have any measurements been made on non-ferrous alloys which have different electrical properties from the steels, but which should obey the same basic laws of fatigue failure?

4. If plastic deformations are detected in a test, these may in some tests be harmful whereas in others, plastic deformations such as those produced by cold rolling are beneficial. Does the method

---

[5] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

employing electrical measurements differentiate between damaging and beneficial types of plastic deformation?

5. Are the "residual stress fields" that are discussed in the paper meant to be magnetic and electrical factors, or can one actually measure mechanical stresses on a micro-scale by means of these electrical measurements?

The field of electrical measurements appears to be the only promising one with which to make investigations to determine damage in fatigue prior to the initiation of a crack. However, we have felt that it is very difficult to correlate random measurements of various types with the actual mechanical phenomena that progress during repeated stressing. Since damage in fatigue may be initiated in a narrow localized region, such as a notch, it would appear difficult to localize the indications read by the cyclograph to the single region of importance.

In connection with the hysteresis losses, I take it that the measurements discussed in the paper were entirely of electrical hysteresis. There has been some interesting work done recently at Syracuse University under B. J. Lazan in which measurements were made of mechanical hysteresis during rotating-beam fatigue tests. The results indicated that the mechanical hysteresis losses increased during all phases of the test, but were decreased by subsequent rest periods. There was no dropping off of hysteresis of the nature shown in the author's electrical measurements during a continued test. The mechanical hysteresis increased very rapidly during the early stage, tended to level off to a fairly constant rate during a second stage, and finally there was a third stage in which the hysteresis increased rather markedly just prior to failure of the specimen. At stress levels as low as 80 per cent of the endurance limit, the hysteresis losses were rather pronounced, and indicated an increase of over 600 per cent in damping capacity. It would be interesting to learn whether these results correlate in any manner with the author's electrical measurements.

MR. JOHN M. LESSELLS.[6]—I believe that this paper by Mr. Duke, which is a continuation of the work of Ellis and Cavanagh, is extremely interesting as a possible tool in studying fatigue phenomena.

In mechanical hysteresis Bairstow[7] and others have shown that the loss is at first large but this tends to decrease and become stable. Whether or not fracture takes place will depend on the stress level.

The information now being made available by the cyclograph seems to indicate certain correlation as regards the magnetic characteristics with the generally accepted concept of mechanical hysteresis.

We are at present continuing this work at Massachusetts Institute of Technology and hope to have additional data at some future date.

MR. JAMES B. DUKE (*author's closure*).—The questions posed by Professor Dolan are both pertinent and interesting, and make it possible to illustrate some points which were not discussed in the body of the paper.

With regard to the differentiation of favorable and unfavorable residual stress fields, the magnetic method will indicate an unfavorable surface stress condition by the sharp reversal in the losses after a few hundred thousand cycles. This reversal is invariably followed by the regular sequences illustrated in the paper. As far as favorable fields are concerned, the levelling of the curve after the original reorientation of the magnetic domains and the establishment of a new magnetic level indicates the presence of fields which have induced a more favorable endurance situation in the specimen, but it does not, so far as I know, indicate the degree of favorable change.

---

[6] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[7] L. Bairstow, *Philosophical Transactions*, Royal Society (1911).

As Mr. Lessells pointed out, there appears to be a direct correlation between mechanical hysteresis and the magnetic effects observed during these tests. From Bairstow's original work, we know that the mechanical hysteresis has an unfortunate tendency to level off, once a plastic state is attained in the material. The Villari reversal in the magnetostriction and the accompanying changes in the relations between stress and the core losses makes the magnetic method more desirable as a fatigue detection instrument. Inasmuch as the Villari reversal is coincident with the inception of fully plastic flow, the magnetic analysis allows investigation of the material during this vital portion of the fatigue life.

So far as I know, very little work has been done with non-ferrous materials using the Cyclograph. This is a very fertile field for future investigations, for the material itself will eliminate one of the most complicating features of the analysis, that of the interdependence of hysteresis and eddy current losses.

The separation of beneficial and harmful forms of plastic deformations in a magnetic analysis occurs only in certain of the specimens tested. With the exception of the determination of favorable residual stress fields as pointed out above, only those specimens which exhibit a second reversal in the losses can be conclusively analyzed. I refer, for example, to Fig. 3 in the paper, and have reference to point *E*. Up to point *E*, little can be said about the lattice destruction in the specimen, but there were cases where the second reversal did not occur. This happened in specimens tested at or near the endurance limit, and from it, I infer that the semi-plastic deformations occurring were of a beneficial nature.

The residual stress fields discussed in the paper are true mechanical micro stress fields, and can be measured on a quantitative basis by the use of a calibrating apparatus described in the paper "Magnetic Stress Analysis" by P. E. Cavanagh and T. Wlodek.[8] Some verification of the residual stress effects described in my paper was obtained by Mr. Wlodek in his doctoral thesis at Massachusetts Institute of Technology. The dynamagnetic apparatus developed by Wlodek is the most promising instrument available for quantitative measurements, either as an independent unit or as a calibrating device.

---

[8] Symposium on Magnetic Testing, Am. Soc. Testing Mats. (1948). (Symposium issued as separate publication, *STH. No. 85*).

AN HYPOTHESIS FOR THE DETERMINATION OF CUMULATIVE DAMAGE IN FATIGUE*

By F. E. Richart, Jr.,[1,2] and N. M. Newmark[1]

Synopsis

It is impossible at present to predict the endurance of a machine part subjected to numerous cycles of different repeated stresses which are above the endurance limit stress. The effect of a number of cycles of one or more overstresses upon the subsequent endurance at another overstress cannot be determined except by test. It is the purpose of this paper to present an hypothesis for the determination of the endurance of a material under any arbitrary overstress-cycle pattern.

A review of the previous work has indicated that it is reasonable to assume the existence of a damage-cycle ratio relation during the process of forming a fatigue crack at any overstress. This relation is different at different stress levels. Various curves on a damage-cycle ratio diagram were assumed to represent the process of failure at different stress levels and were used in computations for endurance under various stress-cycle patterns. As a result of this investigation, it was found that relative *D-R* curves were sufficient to give correct estimates of endurance. An experimental method of determining these relative curves is presented.

Tests were carried out on large plate fatigue specimens and small rotating beam specimens to verify the analysis. The scatter of test points to determine the *S-N* curve made it desirable to use minimum curves. The values of endurance of specimens under several stress-cycle patterns agreed well with the values computed from the experimentally determined *D-R* curves.

The fatigue information usually obtained from samples of a material is given as an *S-N* curve, which is a diagram showing the number of cycles to failure at a constant nominal stress amplitude. From this diagram, only two questions regarding the behavior of the material can be answered. They are:

1. What is the highest stress to which the material may be subjected an infinite number of times?

2. If the stress applied is higher than the endurance limit, how many times must it be repeated to produce failure?

If working stresses above the endurance limit are to be used in design, data from the *S-N* curve can be used directly only when a constant stress amplitude is applied. If the stress amplitude fluctuates during the life of the part, as it actually does in most machine parts, there is no method available at present to estimate the action which a number of cycles of one overstress level has upon the endurance life at another overstress level. It is to

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Special Research Associate in Civil Engineering and Research Professor of Structural Engineering, respectively, Department of Civil Engineering, Talbot Laboratory, University of Illinois, Urbana, Ill.

[2] Now Assistant Professor of Mechanical Engineering, Harvard University, Cambridge, Mass.

be expected that a few cycles of a very high stress has more of a "damaging" action on a specimen than the same number of cycles of a lower stress, since the S-N curve indicates that fewer cycles of a high overstress are available before failure than at a lower overstress. However, the relation between the number of cycles at one overstress needed to produce a given amount of damage, to the number of cycles at a second overstress required to produce the same amount of damage, is at present not clearly defined.

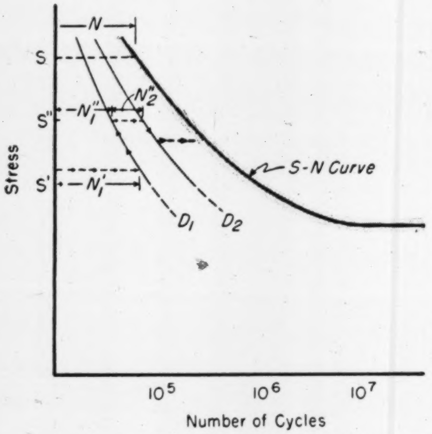


FIG. 1.—Illustration of the Use of "Constant Damage" Lines on the S–N Diagram for Determining Failure Produced by Cycles of Several Stress Levels.

Certain terms are used in this report which require definition or explanation. With reference to the S-N curve shown in Fig. 1, the number of cycles N at a given stress S is called the "endurance life" at that stress. The endurance life at any given stress is actually determined from a "scatter band" of test points (see Fig. 1) and therefore represents a typical or average value. The number of cycles represented by the mean endurance life may vary widely from the value obtained by a single test, but the mean endurance life is statistically adequate. If we consider repetitions of only the stress S, "failure" of the material occurs after N repetitions of stress.

However, failure may be arbitrarily defined, and even the stress S is arbitrary. At the point where failure is initiated, the true maximum stress is by no means a constant for a given constant value of maximum load applied to the test specimen. As the test progresses, changes in properties of the materials and changes in the permanent deformations produced in the specimen introduce changes in the stress distribution. Residual stresses are produced which may make the actual values of maximum and minimum stresses during each cycle considerably different from those we ordinarily naively assume, and the changes in properties may make the range in stress during each cycle differ from the value given by an analysis. For these reasons it is customary to use the so-called "nominal" stress as a basis for studying fatigue phenomena, instead of the actual stress.

As for failure, we may define this as the condition which results in a crack rather than the condition which produces complete rupture of the test specimen or structural or machine part. In this work it is expedient to consider "failure," or "100 per cent damage" as the development of a sizeable crack. Then we may study separately the phenomena which lead to the development or initiation of a visible crack in the material and the phenomena associated with the propagation and spreading of the crack.

It is clear that we can have several S-N curves for the same type of specimen, depending on the definition of failure and the precise means of computing or measuring the stress. But when we have arbitrarily settled on "nominal" stress, and "first appearance of a crack," we have a unique curve for a particular size and shape of specimen made of a given material which is tested under a given repeated stress pattern.

For the given stress S, any number of cycles $N_1 = RN$ corresponds to some "degree of damage" less than unity or 100 per cent damage. R is called the "cycle ratio," and D denotes the "degree of damage." Then as R varies from 0 to 1, D also varies from 0 to 1. (Or both quantities may vary from 0 to 100 per cent.)

Now R is an easily defined quantity, but we have not yet defined what is meant by any degree of damage D less than 100 per cent. This is the point where it is believed that a new concept is introduced and a new and more useful approach is presented to the problem of cumulative damage due to combinations of different intensities of stress.

It obviously makes no practical difference how we define D when we consider only one stress S. However, when we consider several stresses, then we must have a definition which is not ambiguous. That is, the same value of D should apply to the particular number of cycles N_1' at the stress S' which applies to the number of cycles N_1'' at the stress S'' if these two conditions correspond to the same subsequent endurance until failure at any arbitrary stress S. This sort of definition still leaves D arbitrary to some extent, which is a convenience since it may permit a correlation with some other physical property of the material or some separate criterion of damage.

But there is an implicit assumption hidden in the foregoing. This is the implication that a given D reached in a certain way is unique. This may not be the case; the two conditions cited may yield the same subsequent endurance at several different values of S but not at all values. If the assumption is true it will be possible to draw lines of equal D on the S-N plot; the line representing endurance life is only one of the family of curves, for the particular value $D = 1$. Then we can obtain cumulative effects at different stresses by the simple process of locating the point S', N_1' and the value of D, say D_1 which corresponds to this point which represents the effects of N_1' cycles at a stress S'. If this is followed by N_2'' cycles at a stress S'', we proceed along the D_1 curve to the stress S'' and then along the N direction for a distance which corresponds to N_2'' cycles. This brings us to a value of D_2, which is the cumulative damage due to the combination of stresses. We can proceed in this way until we reach a value of $D = 1$, which corresponds to failure. The procedure just described is represented on Fig. 1. It should be noted that the S-N curve (or $D = 1.0$ curve) is the only curve on this diagram which represents *failure* of the specimens. The constant damage curves such as D_1, D_2, etc., do not represent failure, but only a "degree of damage."

For each value of the stress S there must exist some relation between the damage D and the number of cycles applied. This relation can be represented in a dimensionless form as a curve of some type on a D-R diagram. For convenience in the following discussion cycle ratio, R, was chosen as the abscissa and damage, D, was chosen as the ordinate. Studies of various types of curves on this D-R diagram are made in the analytical part of this investigation.

Other terms which are used frequently in the following analysis and should be defined are "blocks of cycle ratio" and "value of cumulative cycle ratio." By blocks of cycle ratio we mean a number of cycles at a given stress which represents a given increment in value of cycle ratio. Cycle ratio has been defined as the ratio of the number of cycles applied at a given stress level to the number of cycles to failure, or the endurance life, at that stress. Thus if we apply 10 per cent cycle ratio at one stress level, then change the stress level and apply 10 per cent cycle ratio of a second stress, for convenience we say that we have applied 10 per cent blocks of cycle ratio at each stress. The value of cumulative cycle ratio is merely the summation of the various blocks of cycle ratio which were applied before failure of the specimen occurred.

Object and Scope of Work:

The purpose of this paper is to attempt to find a method whereby the damaging actions due to cycles of different stress levels can be determined. If it is possible to determine these individual amounts of damage imposed by a number of cycles of each stress level, it would facilitate the prediction of endurance life of the material under any complicated loading pattern.

This investigation is composed of the analytical work and the experimental verification of this analysis.

The analytical work consists of the following: a basic theory for the computation of the value of cumulative cycle ratio obtained to failure using infinitesimal steps on the curves of the $D = R^n$ family; a study of the value of cumulative cycle ratio obtained applying large blocks of cycle ratio to pairs of arbitrarily chosen curves on the cycle ratio *versus* damage diagram; and a method by which relative cycle ratio *versus* damage curves may be determined experimentally.

The experimental portion of this investigation consists of tests to determine these relative cycle ratio *versus* damage curves by the method determined in the analytical work. Tests were carried out on large ($\frac{1}{2}$-in. plate) specimens, containing a 1-in. hole as a stress raiser, which were axially loaded from zero to a maximum tensile load. Tests were also run on 0.100- and 0.150-in. diameter cantilever rotating-beam specimens in a University of Illinois type high-speed fatigue testing machine.

Acknowledgment:

The work reported herein was performed in the Engineering Experiment Station of the University of Illinois, as a part of cooperative projects with both the Office of Naval Research (contract N6ori-71, Task V) and the David W. Taylor Model Basin of the Bureau of Ships (contract NObs-34182), of the Navy Department.

REVIEW OF PREVIOUS WORK

The first significant investigation of the overstress region below the *S-N* curve was reported by French (**2**)[3], who established the existence of a "probable damage line." This probable damage line is valid for cycles of one stress level only and cannot be used for combining the effects of cycles at several stress levels.

Kommers (**4**) has investigated the damaging effects of various numbers of cycles of stresses on rotating-beam fatigue specimens by measuring the reduction of the endurance limit of these damaged specimens. Kommers (**5**), Bennett (**1**), and others have also used the reduction of endurance life as the measure of the damaging effect of the prestress. Bennett proposed a method of estimating endurance of specimens by the use of an experimentally determined "strength-loss" line. However, the determination of this strength-loss line is based on the assumption that the *S-N* curves may be represented as straight lines in the overstress region, but many materials produce curves in this region.

Miner (**6**) has proposed that the life of a specimen or part is governed by a relation of the cycle ratios at the various stress levels such that the summation of the cycle ratios equals unity. Tests performed by Kommers, Bennett, and this investigation have not agreed with Miner's proposal.

---

[3] The boldface numbers in parenthesis refers to the list of references appended to the paper, see p. 798.

The previous investigations have indicated that the damaging effects of a number of cycles of a prestress are indicated in several ways. The reduction of endurance limit and the reduction of endurance life have been used most frequently to indicate the amount of damage sustained. However, in the following discussion, no attempt will be made to correlate or combine the various phenomena which have been used to indicate that damage has occurred. Instead, a new hypothesis is presented which appears to be more useful and simpler to interpret. This hypothesis states briefly that damage to a fatigue test specimen is measured by a quantity D, called "degree of damage," which depends on R, the "cycle ratio," but the dependence is different at different stress-levels.

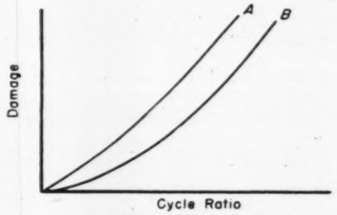
Lines of constant degree of damage D can be constructed on an S-N diagram, and from these the equivalent cycle ratio for any subsequent stress can be determined. However, it is often more convenient to use the dimensionless plot, or the D-R diagram, to represent the relation between damage and cycle ratio.

THEORY FOR DAMAGE ACCUMULATION

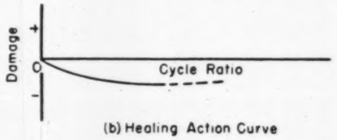
General Remarks:

During a fatigue test several actions take place simultaneously in the material. There is a strengthening or beneficial effect produced by cold working, crystal reorientation, or other changes of this type, while at the same time there is a deleterious action which grows and propagates during the test.

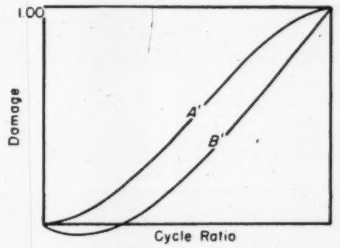
Figure 2 illustrates how these actions combine to produce a single curve of cycle ratio *versus* damage which represents the resultant effect. Two curves, A and B, are shown on Fig. 2(a) which represent the damaging actions which might occur in a specimen when subjected to two different levels of stress. Curve A represents the action at the higher stress since the damage develops more rapidly at the lower values of cycle ratio. The curve of Fig. 2(b) represents the strengthening or "healing" action which occurs. This healing curve lies in the negative damage range of the diagram and since it tends to become horizontal, its influence on the resultant curve is significant only at the lower values of cycle ratio. The resultant curves are shown on Fig. 2(c) as curves A' and B'. In the formation of curve A' it is noted that the damaging effect is at all points predominant so that this curve lies entirely in the positive damage region. On the other hand, for curve



(a) Damaging Action Curves



(b) Healing Action Curve



(c) Resultant Curves = (b)+(a)

FIG. 2.—Formation of the Resultant Cycle Ratio *versus* Damage Curves.

B′ the healing action is predominant for low values of cycle ratio and the curve dips into the negative damage region of the diagram before the damaging effect assumes predominance and eventually causes failure. In case the amount of the damaging action never exceeds the value of the healing action, failure does not occur. However, in this case, the cycle ratio *versus* damage curves are meaningless since 100 per cent cycle ratio equals infinity.

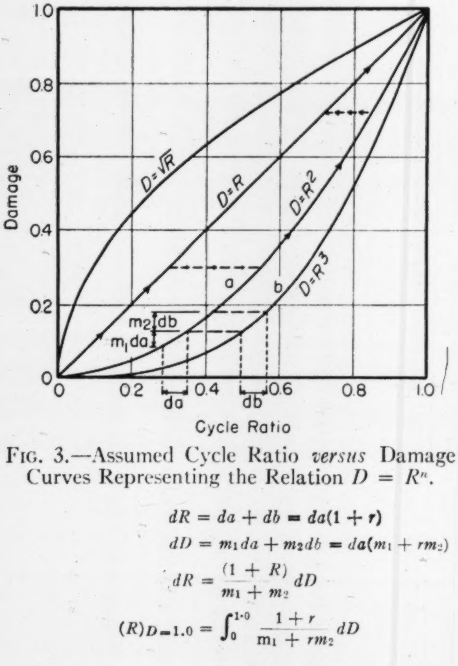


FIG. 3.—Assumed Cycle Ratio *versus* Damage Curves Representing the Relation $D = R^n$.

$$dR = da + db = da(1 + r)$$

$$dD = m_1 da + m_2 db = da(m_1 + rm_2)$$

$$dR = \frac{(1 + R)}{m_1 + m_2} dD$$

$$(R)_{D=1.0} = \int_0^{1.0} \frac{1 + r}{m_1 + rm_2} dD$$

The progress of the action causing failure in a given material at a given stress level may be represented by some individual curve on the cycle ratio *versus* damage diagram. It is important to note that the shape of this curve is dependent on the intensity of stress. In this study various curves on the cycle ratio *versus* damage diagram are assumed and their implications are considered in cases where different intensities of stress are combined in various arbitrary ways.

The $D = R^n$ Family of Curves:

It was convenient to assume the family of curves represented by the equation $D = R^n$ (see Fig. 3) for the initial investigation. Here D denotes damage, R denotes cycle ratio and n the exponent which determines the position of a given curve on the cycle ratio *versus* damage diagram. This diagram includes the ranges of cycle ratio and damage from 0 to 1.0. At a damage of 1.0 it is assumed that the fatigue crack has initiated and thus may not be removed, as by understressing, heat treating, etc. The cycle ratio is 1.0 at a damage of 1.0, by the definition of cycle ratio, therefore the points (1, 1) and (0, 0) must terminate any assumed curve.

The family of curves $D = R^n$ passes through the points (0, 0) and (1, 1) as shown in Fig. 3, in which each curve may represent a different overstress on some material. If one curve is followed from zero damage to 1.0 damage, the total cycle ratio involved is 1.0. This corresponds to a fatigue test using a constant overstress. However, if two curves are used alternately with infinitesimal increments of damage added by each curve, the value of accumulated cycle ratio obtained before failure is less than 1.0. When two curves are used to represent the process of failure in a specimen it represents an action which is the result of two levels of stress applied to the specimen. For instance, the first curve might represent a stress condition of ±30,000 psi. while the second curve represents the stress ±40,000 psi. When we jump from one curve to the other this represents the shift of the stress on the specimen from ±40,000 psi. to ±30,000 psi. or *vice versa*. Furthermore, it is assumed in all further computations and graphical work that the action termed as damage is of the same nature for all stresses. That is, if 50 per cent damage is produced by a certain number of cycles of a given stress and the stress level is changed, the specimen remains damaged 50 per cent until cycles of the second stress level increase or decrease this value of 50 per cent. Thus, all jumps from one curve to another are made along horizontal, or constant damage, lines on the cycle ratio *versus* damage diagrams.

The expression for the value of cumulative cycle ratio obtained before failure is determined as follows:

For the two curves a and b, Fig. 3, we have m_1 as the slope for curve a, m_2 the slope of curve b, and da and $db = r\,da$ as the increments of cycle ratio added. The ordinate gained as a consequence of da cycles of curve a is $m_1\,da$, while that gained due to $db = r\,da$ cycles of curve b is $m_2\,r\,da$. Thus,

$$dR = da + db = da\,(1 + r)$$
$$dD = da\,(m_1 + rm_2)$$

and therefore:

$$dR = \frac{1 + r}{m_1 + rm_2}\,dD$$

or

$$(R)_{D=1.0} = \int_0^{1.0} \frac{1 + r}{m_1 + rm_2}\,dD \,..\,(1)$$

Equation 1 is the general expression for the value of total cycle ratio which may be accumulated by infinitesimal increments of the two curves before failure occurs. The value of r represents the ratio of the cycle ratio increment of curve b applied to the cycle ratio increment of curve a, and even if r is large the assumptions of the method restrict the increments to very small values.

Only one example to illustrate use of this expression will be treated here, since the method is similar for other pairs of curves and involves only simple integration. If we take the two curves, $D = R$ and $D = R^2$, the respective slopes are $m_1 = 1$, $m_2 = 2R = 2\sqrt{D}$. Then placing these values in Eq. 1, we get

$$(R)_{D=1.0} = \int_0^{1.0} \frac{1 + r}{1 + 2r\sqrt{D}}\,dD = \left(\frac{1 + r}{r}\right)\left[\sqrt{D} - \frac{1}{2r}\log_e\,(1 + 2r\sqrt{D})\right]_0^{1.0}$$

$$= \frac{1 + r}{r}\left[1 - \frac{1}{2r}\log_e\,(1 + 2r)\right] \,\ldots\ldots\ldots\ldots\,(2)$$

From Eq. 2, the following values are obtained:

$r = 1.0,\ R_{1.0} = 0.9014$
$r = 0.10,\ R_{1.0} = 0.9724$
$r = 100,\ R_{1.0} = 0.9832.$

By a simple transformation, it can be shown that the same value of $R_{1.0}$ is obtained for the two curves $D = R$ and $D = R^2$ as for any two curves $D = R^n$, $D = R^{2n}$. This is an illustration of the same point which was discussed previously regarding the arbitrary character of the definition of D.

If we wish to compute the value of cumulative cycle ratio to failure, using large blocks of cycle ratio, or if we wish to determine this value for curves represented by complex equations, it is more convenient to use graphical means. That is, the curves must be drawn to a large scale and the corresponding intermediate points determined graphically.

To compute the cumulative damage, or to compute the total per cent cycle ratio which may be applied up to the point of unit damage, it is necessary to utilize two or more curves. Choosing the two curves, $D = R$ and $D = R^2$, the method of computing total cycle ratio to failure by the graphical method may best be illustrated by an example. If 30 per cent cycle ratio is to be added at each stress level alternately to failure, then the value of cumulative cycle ratio depends upon which curve is used first. Using $D = R$ first, 0.30 cycle ratio on $D = R$ produces 0.30 damage; at 0.30 damage, the equivalent cycle ratio on the $D = R^2$ curve is 0.548. Adding 0.30 cycle ratio to 0.548 gives a total cycle ratio on the $D = R^2$ curve of 0.848 which corresponds to 0.719 damage. The equivalent cycle ratio on the $D = R$ curve at 0.719 damage is 0.719, leaving only 0.281 cycle ratio to failure. Thus, the total cumulative cycle ratio is 0.881 before 100 per cent damage. This process just described is shown on Fig. 3 by arrows and the dashed line. Using the $D = R^2$ curve for the first 0.30 block of cycle ratio and continuing in the above manner gives a value of cumulative cycle ratio = 1.044.

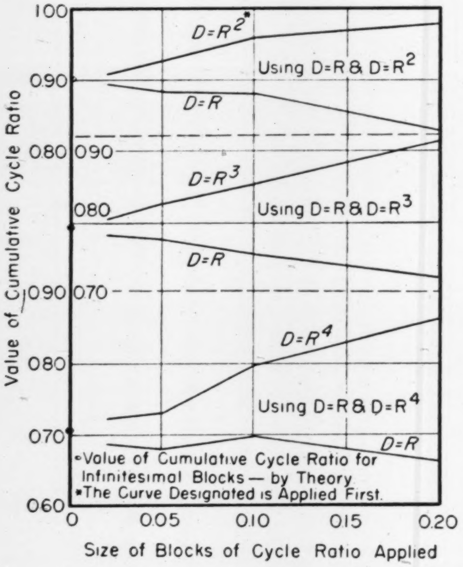
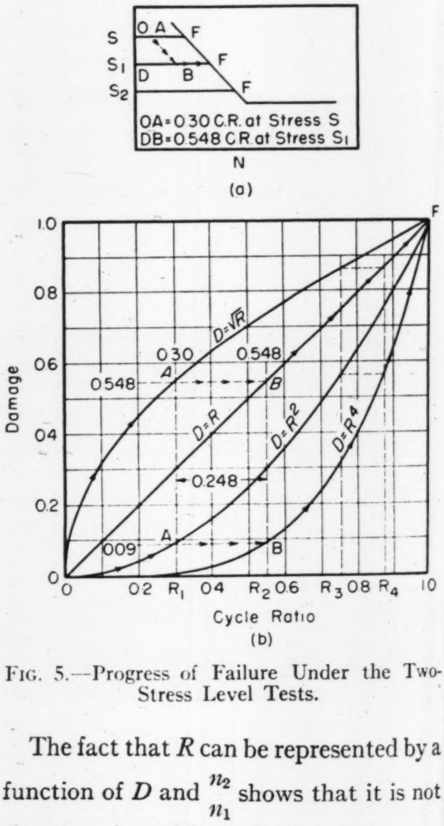


FIG. 4.—Convergence of the Values of Cumulative Cycle Ratio When Small Equal Sized Blocks of Cycle Ratio Are Applied to Curves of the $D = R^n$ Series.

Figure 4 shows that by reducing the size of the applied blocks of cycle ratio, the curves which represent the cumulative cycle ratio to failure, computed using each damage curve first, appear to converge toward a definite value. The value of cumulative cycle ratio converged upon is not constant for all pairs of curves but varies approximately as a straight line with $\frac{n_2}{n_1}$ where $n_2 > n_1$.



(a)

(b)

FIG. 5.—Progress of Failure Under the Two-Stress Level Tests.

The fact that R can be represented by a function of D and $\frac{n_2}{n_1}$ shows that it is not the actual position of the two curves which is significant in the computation of cumulative cycle ratio, but rather it is the relative locations of the two curves. As an example, take the two pairs of curves, $D = \sqrt{R}$, $D = R$; and $D = R^2$, $D = R^4$ both pairs having a value of $\frac{n_2}{n_1} = 2.0$. At a value of cycle ratio of

0.30, the ordinate of $D = \sqrt{R}$ is $\sqrt{0.30} = 0.548$ and the ordinate of $D = R^2$ is $0.30^2 = 0.090$. The corresponding value of cycle ratio on the $D = R$ curve at ordinate 0.548 is 0.548 while the value of cycle ratio on the $D = R^4$ curve at ordinate 0.090 is $\sqrt[4]{0.090} = 0.548$. Thus, the displacement of each pair of curves along the constant damage line is $0.548 - 0.300 = 0.248$ for the initial cycle ratio point of 0.300 on the curve of lower value of n. Figure 5 shows the procedure just described. Both Fig. 5(*a*) and 5(*b*) represent the same procedure by following along the path *OABF* to failure at *F*. Figure 5(*b*) illustrates the proposition that the value of cumulative cycle ratio obtained to failure is the same when any pair of $D = R^n$ curves having a constant value of $\frac{n_2}{n_1}$ is used. The value of cumulative cycle ratio is $0.300 + 0.452 = 0.752$ regardless of whether the path *OABF* is along $D = \sqrt{R}$ and $D = R$ or whether it is along $D = R^2$ and $D = R^4$. The only difference is the level or degree of damage at which the transfer is made from the initial curve to the final curve. This jump from one curve to the other represents a change of stress level on the specimen and the fundamental assumption of this hypothesis is that the subsequent stress cycles either add to or subtract from the degree of damage which was produced by the initial stress cycles.

The preceding paragraph along with Fig. 5 has illustrated that this value of cumulative cycle ratio is the same when large blocks of cycle ratio are used with pairs of curves of like values of $\frac{n_2}{n_1}$. Table I further illustrates this principle and is included to show the tabular form for computations of cumulative cycle ratio that was used throughout this investigation.

The relation between pairs of curves having the same value of $\frac{n_2}{n_1}$ is such that the value of $R_2 - R_1$ for all pairs of curves is constant for a given value of R_1. This is shown clearly on Fig. 5(*b*). The horizontal displacement $R_2 - R_1$ or the distance *AB* is constant for the three pairs of $D = R^n$ curves shown when this displacement is measured from the same value of R_1 starting from the one curve of the pair which has the lower value of n. That is, for the pairs of curves $D = \sqrt{R}$ and $D = R$, $D = R$ and $D = R^2$, $D = R^2$ and $D = R^4$ the horizontal displacement $(R_2 - R_1)$ is 0.248 when the starting point of $R_1 = 0.300$ is used on the $D = \sqrt{R}$, $D = R$, and $D = R^2$ curves respectively for the three pairs.

TABLE I.—COMPUTATION OF CUMULATIVE CYCLE RATIO USING 50 PER CENT BLOCKS OF CYCLE RATIO.

Order of Curves Used	Total Damage	Equivalent Cycle Ratio	Increment Cycle Ratio Added	Equivalent Total Cycle Ratio	Equivalent Damage	Cumulative Cycle Ratio
$D = \sqrt{R}$	0	0	0.50	0.50	0.707	0.50
$D = R$	0.707	0.707	0.293	1.00	1.00	*0.793*
$D = R$	0	0	0.50	0.50	0.50	0.50
$D = \sqrt{R}$	0.50	0.25	0.50	0.75	0.866	1.00
$D = R$	0.866	0.866	0.133	1.00	1.00	*1.133*
$D = R^2$	0	0	0.50	0.50	0.25	0.50
$D = R^4$	0.25	0.707	0.293	1.00	1.00	*0.793*
$D = R^4$	0	0	0.50	0.50	0.0625	0.50
$D = R^2$	0.0625	0.25	0.50	0.75	0.5625	1.00
$D = R^4$	0.5625	0.866	0.133	1.00	1.00	*1.133*

This horizontal displacement relation between these curves indicates that it is not the actual position of these pairs of curves on the *D-R* diagram, but that it is their relative location which is sufficient to determine the value of cumulative cycle ratio from their use. Thus if we are permitted to use relative curves it means that we do not have to determine the actual degree of damage which exists at a given jump from one curve to

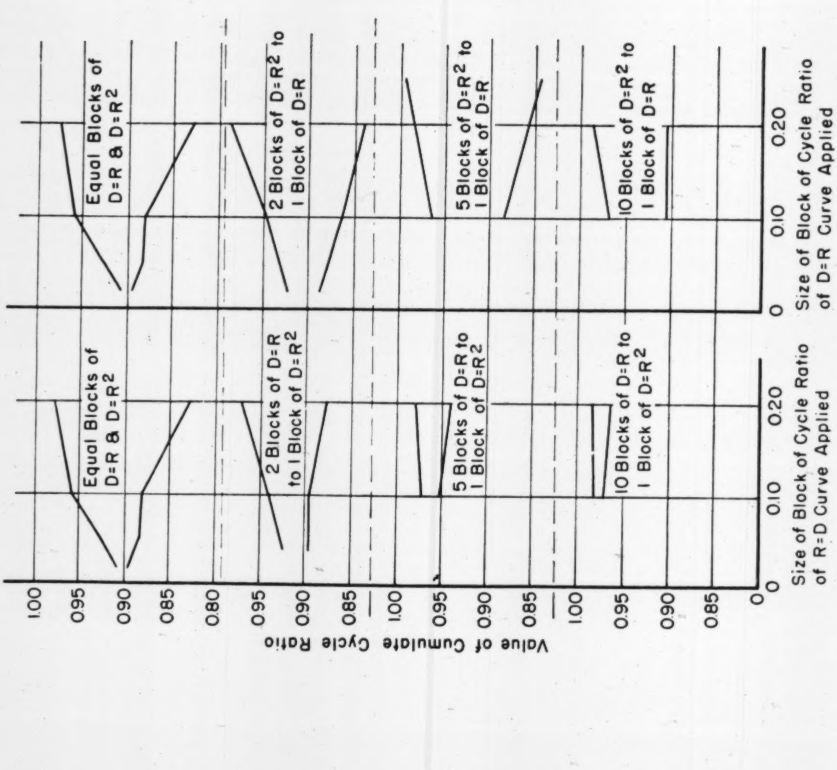


FIG. 6.—Influence of Ratio of Sizes of Blocks of Cycle Ratio Used on the Limiting Value of Cumulative Cycle Ratio.

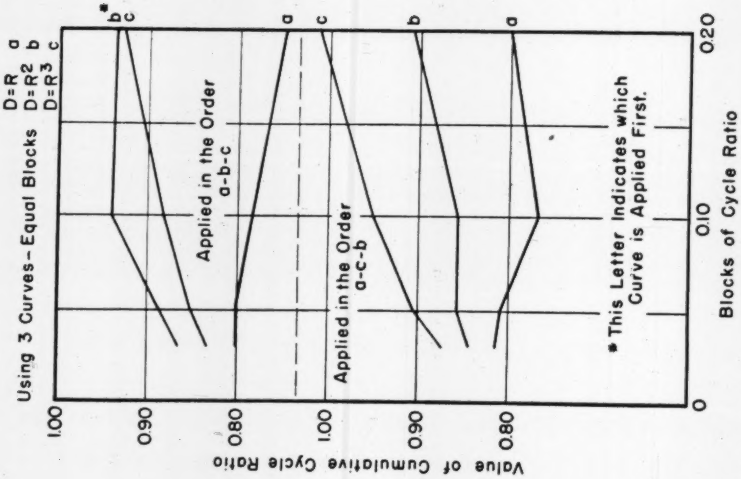
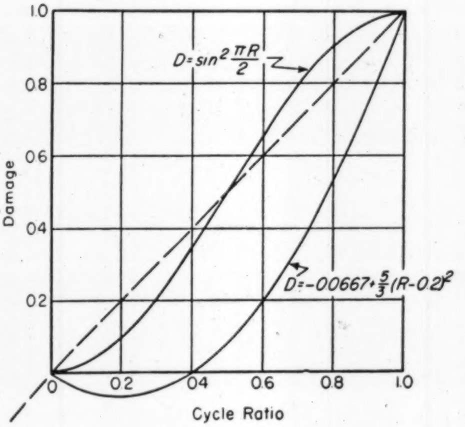
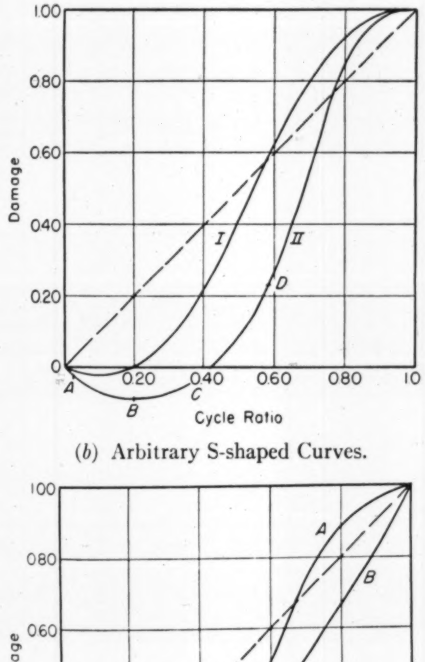


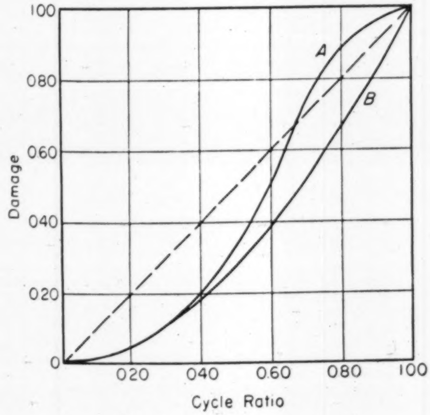
FIG. 7.—Diagram Showing Convergence of the Values of Cumulative Cycle Ratio When Equal Blocks of Cycle Ratio Are Added to Three Curves of the $D = R^n$ Family.



(*a*) **Complex Curves.**



(*b*) Arbitrary S-shaped Curves.



(*c*) **Arbitrary Curves.**

FIG. 8.—Curves Assumed for the Cycle Ratio *versus* Damage Relationship.

another but may use any arbitrary value of D.

The use of relative curves for the determination of cumulative cycle ratio is not restricted to pairs of curves of the $D = R^n$ family but is applicable to any pairs of curves that are so related as to give equal values of $R_2 - R_1$, or horizontal displacement, at every value of R_1. This fact is utilized under the heading "Discussion of Test Results" where the experimental procedure for the determination of the relative curves is described.

The foregoing discussion has considered the computation of values of cumulative cycle ratio only for the condition that equal sized blocks of cycle ratio are applied to each curve. When unequal sized blocks of cycle ratio are used on the $D = R$ and $D = R^2$ curves, the computations indicate that the greater the inequality of the blocks, the closer the value of cumulative cycle ratio converged upon is to 1.0. Figures 4 and 6 indicate that for equal blocks this value is about 0.90; for a 2 to 1 ratio it is about 0.91; for a 5 to 1 ratio it is 0.92 to 0.93; and for a 10 to 1 ratio it appears to be 0.95 to 0.98. Using only one curve, or a ratio of infinity to 1 the maximum value of 1.0 would be obtained.

A more complicated type of loading is represented by using three different curves. Computations to determine the value of cumulative cycle ratio for the three curves, $D = R$, $D = R^2$, and $D = R^3$ show that a value of 0.82 to 0.85 is approached (Fig. 7), regardless of the order in which the curves were used. However, as in all previous computations, when blocks of cycle ratio greater than 1 to 2 per cent are used, the order in which the curves are used is significant. When two curves are used, applying the first cycle ratio block on the curve with the larger value of n (in the Eq. $D = R^n$) produces the larger value of cumulative cycle ratio. For large blocks of cycle ratio, this value may exceed 1.0.

Complex Curves:

Along with the family of curves represented by the equation $D = R^n$, a number of sets of more complex curves were studied. Some of these had regions of "negative damage" and several were made to have an S-shape as shown in Fig. 8. The results from the analyses of these curves showed the same general trends as were found using $D = R^n$ curves. In all cases the values of cumulative cycle ratio obtained with different sizes of blocks showed convergence as the block size decreased, but in several instances the curves representing the different starting points of stress crossed each other several times as they converged. This fact eliminates the possibility of formulating a correlation between the spread at any given block size and the limiting value of cycle ratio for infinitesimal blocks.

TABLE II.—VALUES OF CUMULATIVE CYCLE RATIO TO FAILURE FOR VARIOUS SIZED BLOCKS OF CYCLE RATIO.

The cumulative cycle ratio is given on the line corresponding to the curve for the first stress applied.

Curves Used	Value of Cumulative Cycle Ratio for $r = 1.0$ Size of Blocks of Cycle Ratio Used								Approximate Limiting Value
	0.20		0.10		0.05		0.02		
$D = R$	0.932		0.941		0.953		0.955		0.962
$D = R^{1.5}$		1.009		1.007		0.984		0.973	
$D = R$	0.828		0.880		0.882		0.894		0.901
$D = R^2$		0.978		0.959		0.926		0.907	
$D = R$	0.760		0.790		0.818		0.835		0.846
$D = R^{2.5}$		0.939		0.888		0.871		0.856	
$D = R$	0.719		0.752		0.774		0.781		0.792
$D = R^3$		0.914		0.852		0.824		0.803	
$D = R$	0.664		0.696		0.679		0.686		0.704
$D = R^4$		0.861		0.796		0.729		0.721	
$D = R$	0.625		0.580		0.577		0.628		0.638
$D = R^5$		0.825		0.680		0.627		0.648	
$D = R$	0.875		0.880		0.914		0.923		0.932
$D = \sin^2 \frac{\pi R}{2}$		1.023		0.959		0.956		0.941	
$D = R$	0.706		0.727		0.731		0.745		0.756
$D = -0.6 + 5/3(0.2 - R)^2$		0.951		0.889		0.812		0.768	
$D = R$	0.812		0.809		0.807		0.813		0.820
Curve I		1.011		0.924		0.871		0.834	
$D = R$	0.681		0.638		0.634		0.649		0.660
Curve II		1.001		0.810		0.753		0.671	
$D = R$	0.875		0.903		0.896		0.906		0.916
Curve A		0.988		0.983		0.940		0.926	
$D = R$	0.847		0.846		0.867		0.882		0.890
Curve *B*		0.921		0.935		0.899		0.904	
Curve *A*	0.899		0.962		0.938		0.957		0.951
Curve *B*		1.015		0.942		0.961		0.945	

A summary of the analytical data obtained pertaining to the various *D-R* curves is given in Table II.

Effect of Understresses:

Although the subject of this paper deals primarily with overstresses and the proportions of the total damage contributed by the various levels of overstress, a brief mention of the effects of understresses is included here. The normal operating stresses in most machine parts are below the endurance limit of the material used and thus are understresses, but occasionally overstresses are applied either intentionally or accidentally. Hence in any normal operating cycle there appears a large number of various levels of understress along with a limited number of cycles of overstresses.

If several million cycles of a stress at or just below the endurance limit are applied to a specimen, and then if the stress is raised slightly and again applied for a few million cycles, this process may be repeated numerous times before the specimen fails. This procedure of raising the stress level by small increments after a large number of cycles at each step has been termed "coaxing" and will strengthen the specimen considerably. For some materials it is possible to "coax" the endurance limit to 50 or 60 per cent above its original value. This does not appear to be the case if the subsequent higher stress is considerably larger than the prestress.

It has also been determined that intermediate periods of understress will increase the total number of cycles available at a given overstress. Thus it appears that it is possible for the understress to counteract the damaging effects of the overstress to some degree. Naturally, the amount of "damage" or "healing" produced depends upon the magnitude of these over-and-understresses and the number of cycles of each. If a sufficient number of cycles of the understress is applied to produce a significant amount of "healing," the damage-healing actions during a test may be represented on the cycle ratio *versus* damage diagram as is shown on Fig. 9. Curve *OAB* represents the original cycle ratio *versus* damage relationship of a specimen at a given stress. If at point *A*, the given stress is removed and an understress is applied, healing, or negative damage is produced. This is shown on the figure as a vertical drop to point *C*. The line *AC* is vertical since any given number of cycles of understress produces a value of cycle ratio equal to 0. When the understressing is completed and the given stress reapplied, the curve *CD*, parallel to *AB* is followed until at *D* an understress is again applied. When point *F* (at damage = 100 per cent) is finally reached the value of cycle ratio is much larger than 1.0. By this type of stress pattern, the life of a specimen may be greatly increased at the given overstress.

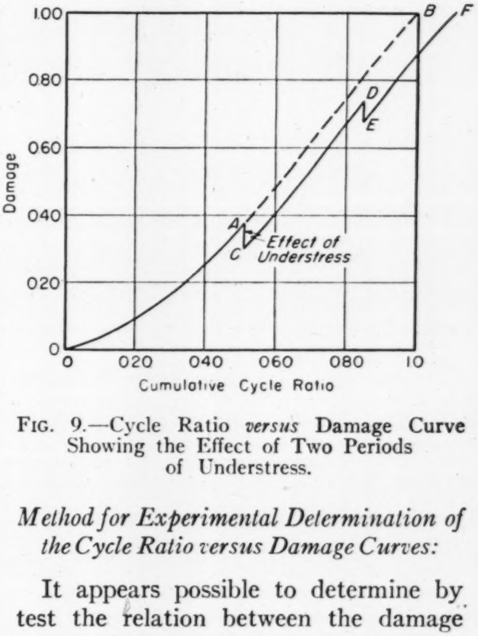


FIG. 9.—Cycle Ratio *versus* Damage Curve Showing the Effect of Two Periods of Understress.

Method for Experimental Determination of the Cycle Ratio versus Damage Curves:

It appears possible to determine by test the relation between the damage curves for several stresses. Since it appears valid that the horizontal difference or maintenance of the cycle ratio relationships between the curves is the significant factor in the computation for cumulative damage, then relative curves are sufficient.

To determine the cycle ratio *versus* damage relation for three different stresses on a given material, the following procedure may be followed. First, select one stress to be used as a prestress and assume any cycle ratio *versus* damage relation such as $D = R$ or $D = \sqrt{R}$ to represent this stress.

Next apply various cycle ratios (20 per cent, 40 per cent, etc.) of this prestress to a series of specimens, then test some of these specimens to failure at each of the other two stresses. The value of cycle ratio remaining at each stress is subtracted from 1.0 and the difference plotted at the value of damage corresponding to the given cycle ratio of prestress. As an example, on Fig. 10 for 20 per cent cycle ratio of the prestress, 60 per cent cycle ratio remained at stress No. 1 and 50 per cent cycle ratio remained at stress No. 2. Therefore, values of 40 per cent cycle ratio for stress No. 1 and 50 per cent cycle ratio for stress No. 2 were plotted at the value of 20 per cent damage which corresponded to 20 per cent cycle ratio of the prestress. In this manner the cycle ratio relations between the three curves may be determined although their true shape or position on the *D-R* diagram may be still unknown.

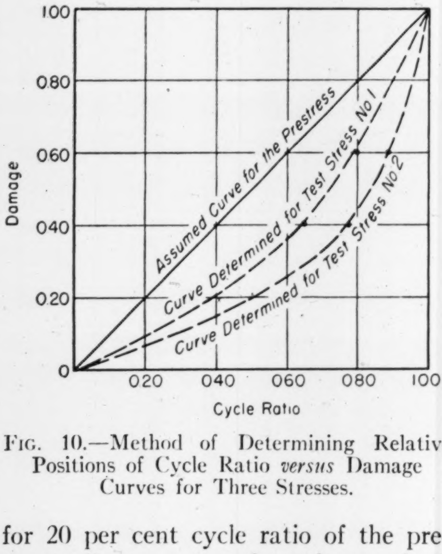


FIG. 10.—Method of Determining Relative Positions of Cycle Ratio *versus* Damage Curves for Three Stresses.

This experimental procedure just described also produces the information necessary for the construction of the "constant damage" lines on the *S-N* diagram as shown on Fig. 1 and Fig. 5(*a*). To illustrate the method of determining line *AB* which connects points having the same degree of damage at the two stresses *S* and S_1 on Fig. 5(*a*) we shall use the example shown on Fig. 5(*b*). For this particular two-stress level test the applied cycle ratio at the prestress *S* was 0.300 and the cycle ratio remaining to failure at the test stress S_1 was 0.452. Then at the stress *S* we establish the point *A* as the distance $OA = 0.300$ cycle ratio. To determine point *B* at the stress S_1, we must measure in a direction of decreasing cycles, or to the left, from the point *F* on the *S-N* curve which represents failure. Thus we lay off $FB = 0.452$ cycle ratio. In this manner we determine two points at different stress levels which represent the same degree of damage to the specimen and by subsequent tests we can establish curves for all overstresses and for as many values of damage as is desired. Of course these values of damage determined in this way do not represent the true value of the damage to the specimen, but they do represent relative values of damage which are sufficient to maintain the proper cycle ratio relationships at the various stress levels.

It should be noted, however, that any discussion which involves use of values based on the endurance life at any stress involves inaccuracies. The endurance life of a specimen is actually defined by a scatter band rather than by a point. Thus, when we assume a curve such as the $D = R$ curve on the cycle ratio *versus* damage diagram for use in the procedure just mentioned, we ignore the scatter involved in the endurance life at that stress. Then when we run tests to determine the two curves relative to the $D = R$ curve, these tests involve scatter bands for all of the endurance lives of the stresses used, and the test points representing the curve for stress No. 1 and stress No. 2 have more than the usual scatter.

After experimentally determining the *D-R* curves or "scatter bands," these may be used in subsequent computation for the value of cumulative cycle ratio or total cycles to be expected under any desired stress-frequency distribution pattern. If the scatter band is wide, it would be advisable to use the worst curve, or the edge of the scatter band curve which forms the greatest difference between itself and the assumed prestress curve. Using these curves of maximum difference, the computations should produce an envelope curve showing the minimum expected value of cumulative cycle ratio. This minimum value or envelope would then be used in subsequent design considerations.

EXPERIMENTAL VERIFICATION OF THE ANALYSIS

Object of Experimental Work:

The object of the experimental work was to prove or disprove the usefulness of the methods indicated in the analysis for experimentally determining these *D-R* curves and for their subsequent use in computing the value of cumulative cycle ratio for various stress-cycle patterns. Tests were run to determine the usual *S-N* curves, the *D-R* curves, and the values of cumulative cycle ratio obtained from using blocks of cycle ratio of about 10 per cent and about 20 per cent. These values of cumulative cycle ratio obtained experimentally were to be compared with those values as determined by the method of the analyses using the *D-R* curves.

The main series of tests were run on large plate specimens which were axially loaded cyclically from zero to maximum tension. Two auxiliary series of tests were run on small rotating-beam specimens to provide additional information.

Description of Specimens:

Two types of fatigue specimens were used in these tests. The main part of this experimental work was carried out using large specimens, machined from $\frac{1}{2}$-in. structural steel plate, having a $3\frac{1}{2}$-in. wide test section which contained a 1-in. diameter hole as a stress raiser. It was hoped that such a specimen would show less scatter of endurance life than smaller specimens usually showed. Also it was desirable to have a large test section so that the area representing a detectable crack would not be a significant portion of the total net area. The 1-in. diameter hole was used as a stress raiser to localize the failure and make detection of the crack easier. The appearance of a definite fatigue crack was assumed to represent failure of the specimen, and all test results are on this basis.

Small rotating-beam specimens were made from structural steel and also from a bar of S.A.E. 4340 electric furnace steel. These specimens were run in a high-speed fatigue testing machine so that considerable additional information could be obtained in a short time.

The large plate specimens and the first series of small rotating-beam specimens were cut from the same $\frac{1}{2}$-in. plate of A-7 killed structural steel conforming to A.S.T.M. Specification A 7 - 46.[4] After rough machining, but before finishing, all specimens of this steel were normalized at 1650 F. for $\frac{1}{2}$ hr. This was to relieve welding and machining stresses and to equalize any grain size variation which might have occurred during the rolling process.

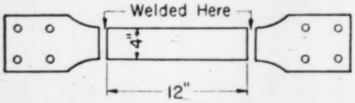
The second series of small rotating-beam specimens were machined from a

---

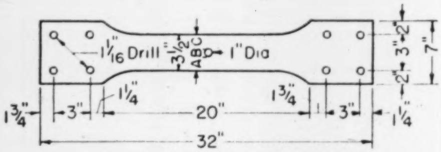
[4] Standard Specifications for Steel for Bridges and Buildings (A 7-46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 1.

TABLE III.—TENSION TEST RESULTS FOR THE A-7 AND S.A.E. 4340 STEELS.

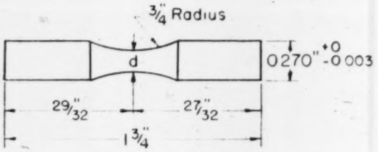
Material	Type Specimen	Yield Point, psi.		Tensile Strength, psi.	Elongation, per cent
		Upper	Lower		
A-7 Steel (RK—Plate N)	0.375 in. diameter	42 600	39 800	64 200	27.7 in 2 in.
	A.S.T.M. Std. Flat	38 200	37 300	63 300	24.6 in 8 in.
S.A.E. 4340	0.505 in diameter	142 000		150 000	18.2 in 2 in.



(a) Fabrication of Large Plate Specimens by Welding Blanks to the Pulling Heads.



(b) Dimensions of the Finished Plate Specimens.



(c) Dimensions of the Small High Speed Specimens

d=0.160" for A-7 Specimens
d=0.105" for EF 4340 Specimens

FIG. 11.—Dimensions of Test Specimens.

3-in. diameter bar of S.A.E. 4340 steel. Specimens were roughed out into $\frac{3}{8}$-in. diameter cylinders before heat treating. These cylinders were heat treated at 1525 F. for 1 hr. 45 min., then quenched in oil. They were then tempered at 1010 F. for 1 hr. 35 min. and finish machined. The chemical composition of this steel was as follows:

Carbon, per cent	0.385
Manganese, per cent	0.780
Phosphorus, per cent	0.010
Sulfur, per cent	0.018
Nickel, per cent	1.80
Chromium, per cent	0.74
Molybdenum, per cent	0.25

Tension tests of samples of both steels were available, and typical results are given in Table III.

The large plate specimens were fabricated by welding 12 by 4 in. blanks, cut from the parent plate, to pulling heads as shown in Fig. 11(*a*). In this manner only a limited amount of pedigreed steel was used in each specimen. After these specimens were welded and heat treated, they were finish machined to the dimensions shown in Fig. 11(*b*). The 1-in. diameter hole at the test section was formed by drilling $\frac{1}{64}$-in. undersize and reaming to size. The surface of the hole was then polished with emery-coated cardboard cylinders, rotated by an electric drill. This polishing removed all scratches or tool marks in the direction of the thickness of the plate and provided a reproducible surface finish. Also the smooth surface was an aid in crack detection since an observer could readily detect a very small crack on this surface, with the aid of a low-power magnifying glass.

The small high-speed specimens were finish machined after heat treatment to the dimensions shown in Fig. 11(*c*). The reduced section was polished longitudinally with No. 00 emery paper and oil to remove all surface scratches and to provide uniform surface conditions for the specimens. All the polishing was done by one man to insure uniformity of the results. This type specimen was cut from both the A-7 and the S.A.E. 4340 steel.

Description of Testing Machines:

The experimental work to substantiate the preceding hypothesis utilized two rather dissimilar types of fatigue testing machines. The large plate specimens were tested in two axial load machines of ±50,000-lb. capacity, while the small rotating cantilever beam specimens were tested in a University of Illinois high-speed cantilever fatigue testing machine.

The 50,000-lb. machines applied an axial load to the specimen by means of a lever attached to the specimen at one end and actuated by a motor-driven eccentric at the other. The range of loads applied to the specimen was adjustable and could be changed by varying the throw of the eccentric, while the maximum or test load was set by a turnbuckle in the connection between the eccentric and loading lever. Applied loads were measured by reading the deflections of a calibrated dynamometer ring.

These large machines are located in pairs on a rectangular framework of heavy I-beams in such a manner that the crank end of one machine is at the same end of the rectangle as the loading end of the other machine. Figure 12 shows one end of this rectangle and includes the eccentric, dynamometer ring, indicating dial, and loading lever of machine No. 8 as well as the pivot post, loading lever, and a specimen in place in machine No. 9.

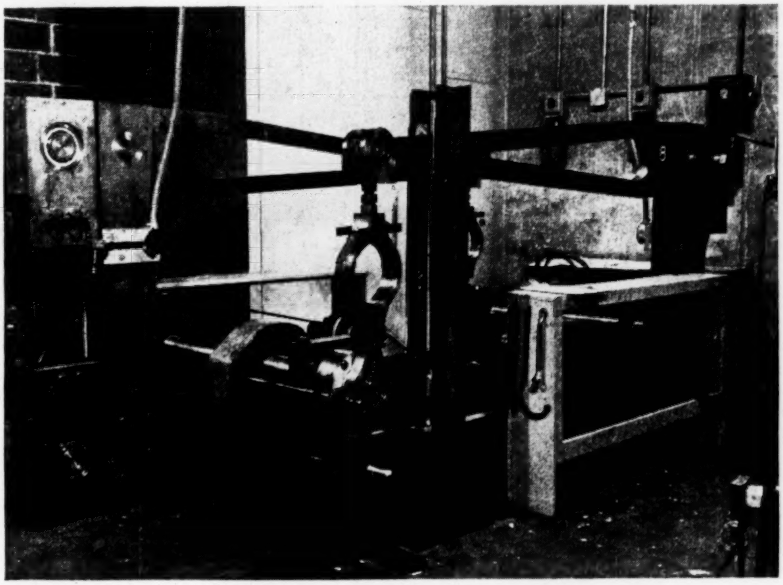


FIG. 12.—Photograph of the Ends of Machines Nos. 8 and 9.

The maximum range of load of these large machines was from 50,000 lb. tension to 50,000 lb. compression, but for these tests the load varied from zero load to a maximum of about 40,000 lb. tension only. The frequency of loading was constant for all ranges of load used and was about 330 cycles per minute or 20,000 cycles per hour. Since these machines were of the "constant strain" type, they required frequent adjusting during each test; consequently, these tests were run only during working hours and were interrupted at night.

The high speed fatigue machine operated at 10,000 rpm. and tested a small specimen of less than 0.250 in. in diameter. An electric motor rotated the assembled cantilever beam which contained the test section of the specimen as its critical section. Two Jacobs chucks held the specimen in place; one chuck was attached to the motor shaft and the other carried a shaft extension which had an enclosed bearing at its outer end. Load was applied to the bearing housing by adjusting the position of a weight along a simple beam which used this housing as one of its supports. The beam was calibrated in inch-pounds so that by adjusting the position of the weight a maximum of 16 in-lb. or a minimum of about 3 in-lb. could be applied to the test section. For the test series using small specimens of A-7 steel, a fatigue machine was used which carried an additional loading device so that, for some tests, up to 21.5 in.-lb. were applied to the specimen. These high-speed machines were of the "constant stress" type and therefore required no adjustment during each test. Each test ran continuously, except when it was necessary to stop it to change the load.

Testing Procedures:

The tests of the large plate specimens were performed over a period of nearly a year. During this time several procedures were adopted to help reduce the "scatter" of the test results. All of these procedures were established during the first series of tests to determine the *S-N* curve and were then used in the following two test series.

The dynamometer rings had been calibrated independently a few months before the beginning of these tests. However, during the first few tests, a "machine factor" appeared, in that the test results obtained in machine No. 8 showed a longer life than those from machine No. 9 at the same stresses. SR-4 electric strain gages were applied to a specimen which was used to calibrate the two machines against each other. As a result of this cross-calibration, the dynamometer constant of machine No. 8 was changed slightly and the machines then checked load indication within 0.5 per cent. All previous data obtained using machine No. 8 was modified as a result of the calibration.

The loads on the specimens in these machines were set by adjusting the throw of the eccentric. The loads had to be adjusted manually before each test and also quite often during any given test. The time required to adjust the loads depended entirely upon the skill of the operator.

The large plate specimens were considered to have failed when a detectable fatigue crack appeared. During the latter part of any given test, numerous inspections were made to discover any such crack. Since the inside surface of the 1-in. diameter hole had received a high polish, it was easy to detect a crack about $\frac{1}{16}$ in. long, visually, when using a low-power magnifying glass. Whenever there was a question about a discoloration on the surface, a version of the "oil and whiting" method was used to determine the true fatigue crack. A tensile load was applied to the specimen and a drop of machine oil was rubbed over the spot, then wiped off. If the discoloration was a true fatigue crack, oil would be forced out to the surface as a small bubble upon release of the load on the specimen. The whiting was not needed in this case since the surface was polished. By this method cracks $\frac{1}{16}$ in. long were easily identified.

No special procedures were necessary with the small fatigue machine except to avoid bending the specimens during the loading process. A little care was re-

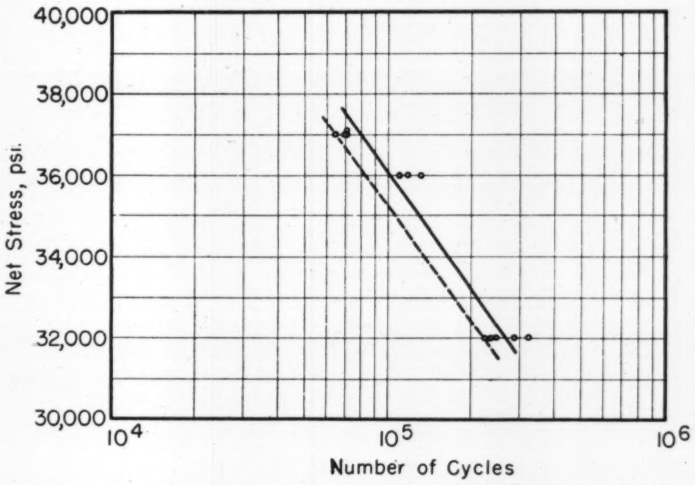


FIG. 13.—*S-N* Diagram for the Large Plate Specimens of A-7 Steel.

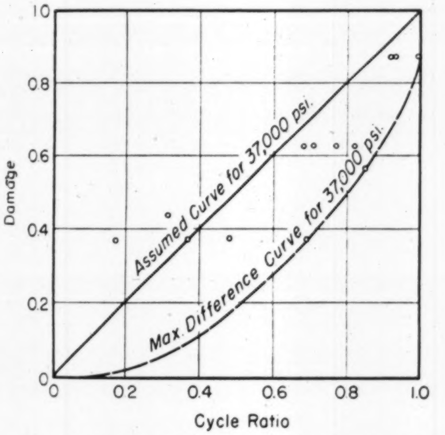


FIG. 14.—Results of Tests of Large Plate Specimens Using 37,000 psi. as Prestress and 32,000 psi. as Test Stress. (Using Values of Cycle Ratio from Average *S–N* Curve.)

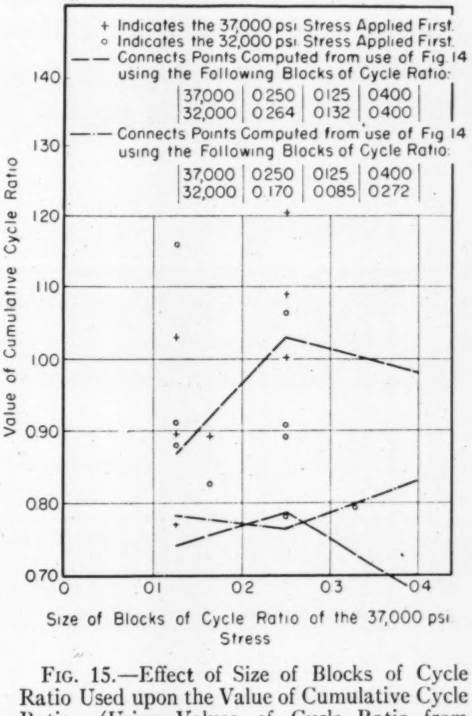


FIG. 15.—Effect of Size of Blocks of Cycle Ratio Used upon the Value of Cumulative Cycle Ratio. (Using Values of Cycle Ratio from Average *S-N* Curve.)

quired to align the specimens in the Jacobs chuck properly and to ease the load onto the bearing housing as the test started. The multiple load tests required that the machine be stopped several times during the test to change the load. During the deceleration period, the load was removed and the outer bearing held to prevent excess vibration which might bend the specimen.

The methods of running the three different series of tests were similar for both the large and small specimens. To determine the *S-N* curve, a given repeated stress was applied to the specimen until failure occurred. The number of cycles or the number of times this stress was repeated gave one point on the *S-N* curve. Numerous tests of this type were necessary to establish the complete *S-N* curve shown in Fig. 13.

The tests to determine the relative *D-R* curves were of the two-stress level or prestress - test stress type. A given number of cycles was applied to specimens at one stress level as a prestress, then these specimens were tested to failure at the second, or test stress level. By varying the number of cycles of prestress, the complete *D-R* curves could be determined by the method previously indicated (5). Some of the results are indicated in Fig. 14.

The alternate block type of test was performed by applying equal sized blocks of cycle ratio alternately at two different stress levels until failure occurred. In the tests of the large specimens, for instance, 20 per cent cycle ratio was applied at 37,000 psi., then 20 per cent cycle ratio was applied at 32,000 psi., and this process continued to failure. Summing up the blocks of cycle ratio applied to failure gave the value of cumulative cycle ratio. By this type of test, information was obtained for the value of cumulative cycle ratio as a function of the size of blocks of cycle ratio, and the results are shown in Fig. 15.

Discussion of Test Results

Tests of The Large Plate Specimens of A-7 Steel:

The results of the large plate specimens are given in Tables IV through VII and are also shown graphically on Figs. 13 through 20. The *S-N* diagram for these specimens is given on Fig. 13. The complete *S-N* curve and the endurance limit were not completely established due to the long time required to run 10,000,000 cycles in the large fatigue machines. The heavy curve shown in Fig. 13 represented the average or typical values for the *S-N* curve while the light line represented the minimum values. The *D-R* curves and the value of cumulative cycle ratio *versus* size of blocks of cycle ratio curves were determined, using both the average curve and the minimum curve of the *S-N* diagram. The first few tests to determine the *S-N* curve showed considerable scatter. It was thought that this was due to "aging" of the steel since some of the specimens had been made six months prior to these tests. The tests to determine the effects of aging showed that there were no aging effects but that there might be some residual stresses present. All subsequent specimens were normalized and much of the scatter of test results then disappeared.

TABLE IV.—RESULTS OF TESTS TO DETERMINE THE *S-N* CURVE FOR LARGE PLATE SPECIMENS.

Specimen No.	Net Stress, psi.	Cycles to First Crack
RK-45	37 100	71 100
RK-52	37 000	70 000
RK-82	37 000	64 300
RK-79	37 000	70 000
RK-18	36 000	110 000
RK-14	36 000	131 500
RK-41	36 000	118 600
RK-50	33 000	170 000
RK-51	33 000	183 200
RK-48	32 000	235 800
RK-49	32 000	226 100
RK-23	32 000	324 900
RK-26	32 000	246 700
RK-27	32 000	288 800

TABLE V.—ENDURANCE LIVES DETERMINED FROM FIG. 25.

Stress, psi.	Average Endurance Life A	Minimum Endurance Life M
37 000	80 000	64 000
34 500	146 000	120 000
32 000	265 000	225 000

The two net stresses of 37,000 psi. and 32,000 psi. were selected for use in

TABLE VI.—RESULTS OF TESTS TO DETERMINE CYCLE RATIO *VERSUS* DAMAGE CURVES FOR LARGE PLATE SPECIMENS.

Specimen No.	Pre-stress, psi.	Total Cycles Prestress	Test Stress, psi.	Total Cycles Test Stress	Using Avg. *S-N* Curve[a]			Using Min. *S-N* Curve[a]		
					C. R. P. S.	C. R. T. S.	Cum. C. R.	C. R. P. S.	C. R. T. S.	Cum. C. R.
RK-58	37 000	70 000	32 000	18 100	0.875	0.068	0.943	1.093	0.081	1.174
RK-64	37 000	70 000	32 000	20 500	0.875	0.078	0.953	1.093	0.091	1.184
RK-79	37 000	70 000	...	0	0.875	0	0.875	1.093	0	1.093
RK-82	37 000	64 300	...	0	0.804	0	0.804	1.000	0	1.000
RK-54	37 000	50 000	32 550	60 100	0.625	0.227	0.852	0.781	0.295	1.076
RK-59	37 000	50 000	32 000	76 800	0.625	0.290	0.915	0.781	0.342	1.123
RK-75	37 000	50 000	32 000	47 400	0.625	0.179	0.804	0.781	0.211	0.992
RK-76	37 000	50 000	32 000	84 200	0.625	0.318	0.943	0.781	0.374	1.155
RK-69	37 000	45 300	32 000	39 500	0.566	0.149	0.715	0.708	0.176	0.884
RK-71	37 000	35 000	32 000	181 600	0.438	0.686	1.124	0.547	0.808	1.355
RK-66	37 000	30 300	32 000	136 600	0.379	0.516	0.895	0.473	0.607	1.080
RK-55	37 000	30 000	32 000	218 600	0.375	0.825	1.200	0.469	0.971	1.440
RK-63	37 000	30 000	32 000	81 400	0.375	0.307	0.682	0.469	0.362	0.831
RK-62	37 000	30 000	32 000	165 800	0.375	0.626	1.001	0.469	0.736	1.205
RK-73	37 000	15 000	32 000	296 600*	0.188	1.12—No Failure		0.234	1.3—No Failure	
RK-70	37 000	70 000	34 500	7 700	0.875	0.053	0.928	1.093	0.064	1.157
RK-81	37 000	70 000	34 500	18 400	0.875	0.126	1.001	1.093	0.153	1.246
RK-68	37 000	50 000	34 500	50 100	0.625	0.343	0.968	0.781	0.417	1.198
RK-84	37 000	50 000	34 500	17 100	0.625	0.117	0.742	0.781	0.143	0.924
RK-85	37 000	50 000	34 500	19 800	0.625	0.136	0.761	0.781	0.165	0.946
RK-67	37 000	30 500	34 500	143 400	0.381	0.982	1.363	0.477	1.193	1.670
RK-72	37 000	30 000	34 500	247 400	0.375	1.693	2.068	0.469	2.060	2.529
RK-53	32 000	157 500	37 000	54 800	0.595	0.685	1.280	0.700	0.855	1.555
RK-60	32 000	157 500	37 000	103 400	0.595	1.293	1.888	0.700	1.618	2.318
RK-56	32 000	112 500	37 000	134 900	0.425	1.685	2.110	0.500	2.110	2.610
RK-65	32 000	112 500	37 000	102 300	0.425	1.280	1.705	0.500	1.600	2.100
RK-57	32 000	67 500	37 000	65 300	0.255	0.816	1.071	0.300	1.020	1.320
RK-61	32 000	67 500	37 000	38 400	0.255	0.480	0.735	0.300	0.600	0.900
RK-74	34 500	105 000	37 000	64 300	0.720	0.804	1.524	0.875	1.000	1.875
WK-77	34 500	105 000	37 000	10 000	0.720	0.125	0.845	0.875	0.156	1.031
RK-78	34 500	75 000	37 000	29 000	0.514	0.362	0.876	0.625	0.453	1.078
RK-80	34 500	75 000	37 000	27 200	0.514	0.340	0.854	0.625	0.425	1.050

C. R. = cycle ratio. P. S. = prestress. T. S. = test stress. Cum C. R. = Cumulative cycle ratio.

TABLE VII.—RESULTS OF TESTS OF LARGE PLATE SPECIMENS USING ALTERNATE BLOCKS OF CYCLE RATIO.

Specimen No.	Size Blocks 10^3 Cycles		First Block psi.	Total Cycles at 37 000 psi.	Total Cycles at 32 000 psi.	Using Average S-N Curve (A-Table)[b]			Using Minimum *S-N* Curve (M-Table)[b]			Size Blocks Average Curve		Size Blocks Minimum Curve	
	at 37,000 psi.	at 32,000 psi.				C.R. at 37 000 psi.	C.R. at 32 000 psi.	Cum. C.R.	C.R. at 37 000 psi.	C.R. at 32 000 psi.	Cum. C.R.	37 000 psi.	32 000 psi.	37 000 psi.	32 000 psi.
RK-28	20	70	37 000	54 500	140 000	0.681	0.528	1.209	0.851	0.622	1.473	0.250	0.264	0.312	0.311
RK-30	20	70	37 000[a]	41 100	138 900	0.673	0.661	1.334	0.840	0.793	1.633	0.328	0.333	0.408	0.400
RK-35	20	70	37 000	45 300	142 000	0.467	0.536	1.003	0.708	0.631	1.339	0.250	0.264	0.408	0.400
RK-86	20	45	37 000	60 000	90 000	0.750	0.340	1.090	0.938	0.400	1.338	0.250	0.170	0.408	0.200
RK-37	10	35	37 000[a]	30 000	84 000	0.492	0.400	0.892	0.612	0.480	1.092	0.164	0.167	0.204	0.200
RK-40	10	35	37 000	40 000	140 300	0.500	0.530	1.030	0.625	0.624	1.249	0.125	0.132	0.156	0.155
RK-42	10	35	37 000	40 000	105 000	0.500	0.396	0.896	0.625	0.467	1.092	0.125	0.132	0.156	0.155
RK-88	10	22.5	37 000	40 000	71 500	0.500	0.270	0.770	0.625	0.318	0.943	0.125	0.085	0.156	0.100
RK-31	20	70	32 000[a]	20 000	119 400	0.328	0.569	0.897	0.408	0.683	1.091	0.328	0.333	0.408	0.400
RK-32	20	70	32 000	40 000	149 100	0.500	0.563	1.063	0.625	0.663	1.288	0.250	0.264	0.312	0.311
RK-34	20	70	32 000	20 200	139 800	0.253	0.528	0.781	0.316	0.621	0.937	0.250	0.264	0.312	0.311
RK-36	20	70	32 000	28 800	140 900	0.360	0.532	0.892	0.450	0.626	1.076	0.250	0.264	0.312	0.311
RK-37	20	45	32 000	40 000	107 500	0.500	0.406	0.906	0.625	0.478	1.103	0.250	0.170	0.312	0.200
RK-38	10	35	32 000	30 600	140 000	0.382	0.529	0.911	0.478	0.623	1.101	0.125	0.132	0.156	0.155
RK-39	10	35	32 000[a]	20 000	105 000	0.328	0.500	0.828	0.408	0.600	1.008	0.164	0.167	0.204	0.200
RK-43	10	35	32 000	40 000	175 000	0.500	0.661	1.161	0.625	0.778	1.403	0.125	0.132	0.204	0.200
RK-89	10	22.5	32 000	40 000	101 000	0.500	0.381	0.881	0.625	0.449	1.074	0.125	0.085	0.204	0.100

---

[a] The stresses applied to these specimens were 33000 psi and 38 100 psi

[b] C. R. = cycle ratio. Cum. C. R. = cumulative cycle ratio.

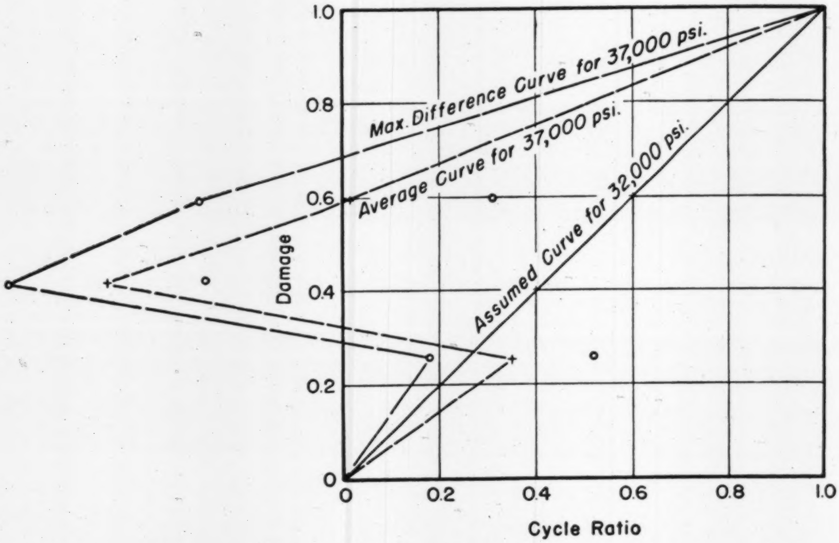


FIG. 16.—Results of Tests of Large Plate Specimens Using 32,000 psi. as Prestress and 37,000 psi. as Test Stress. (Using Values of Cycle Ratio from Average *S-N* Curve.)

the subsequent two-stress level tests. Net stresses, or the total load divided by the net area, were used throughout the tests of these large plate specimens. The stresses at the edge of the hole were above the yield point, and since the material in this region yielded upon first application of the load, the stress distribution was considerably changed from that which could be predicted by the theory of elasticity. Inasmuch as the actual distributions of stress throughout the test section had no effect on the verification of this method of estimating endurance of the specimen under multiple stresses, the peak stress values were ignored and the nominal net stresses were used instead.

The test results for the two-stress level or prestress - test stress type of tests are given in Table VI and in Figs. 14, 16, 17, 19, and 20. The first set of these tests used 37,000 psi. as the prestress and 32,000 psi. as the test stress. These results are shown in Figs. 14 and 17 with the assumed curve for the 37,000 psi. stress and the experimentally determined maximum difference curve for 32,000 psi. This maximum difference curve was used in subsequent calculations for the value of cumulative cycle ratio, since its use gave the expected minimum curves on the value of cumula-

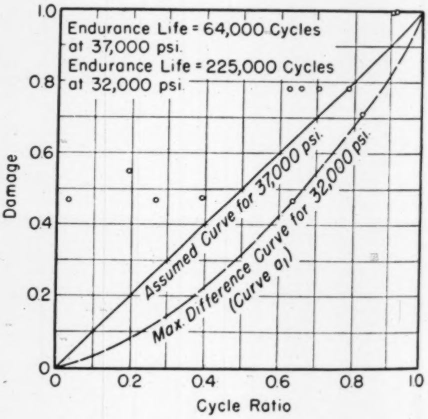


FIG. 17.—Results of Tests of Large Plate Specimens Using 37,000 psi. as Prestress and 32,000 psi. as Test Stress

tive cycle ratio *versus* size of block of cycle ratio diagram.

When 32,000 psi. was used as the prestress and 37,000 psi. as the test stress, the results did not form a smooth curve of maximum difference. The maximum difference curve on Fig. 16 represented the *least* reduction of endurance life at the test stress which was higher than the prestress. This was not a conservative curve. In Fig. 14 where the test stress was lower than the prestress, the maximum difference curve represented the *most* reduction of endurance life at the test stress, caused by the damaging effects of the prestress, and hence was conservative. The use of the maximum difference curve of Fig. 14 in the method described in the analysis gave minimum value curves on the value of cumulative cycle ratio *versus* size of block of cycle ratio diagram which agreed well with the minimum values obtained from tests. For the large plate specimens, no test points fell below this minimum curve (See Figs. 15 and 18).

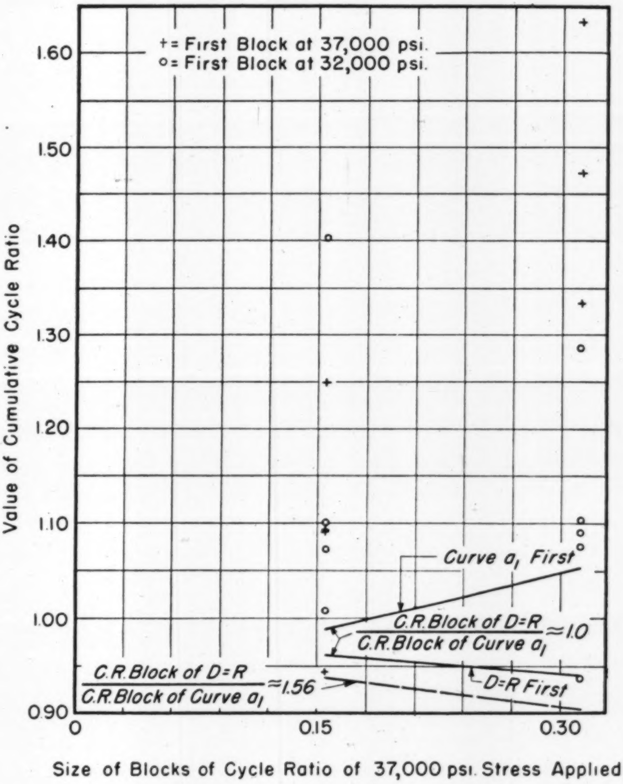


FIG. 18.—Effect of Sizes of Blocks of Cycle Ratio Used upon Value of Cumulative Cycle Ratio.
Endurance Life = 64,000 Cycles at 37,000 psi.
Endurance Life = 225,000 Cycles at 32,000 psi.

These test results for the large plate specimens were computed using endurance lives based on the average *S-N* curve and the minimum *S-N* curve. Thus many of the tests results based on the average, and most of the test results based on the minimum *S-N* curve, indicated cumulative cycle ratios greater

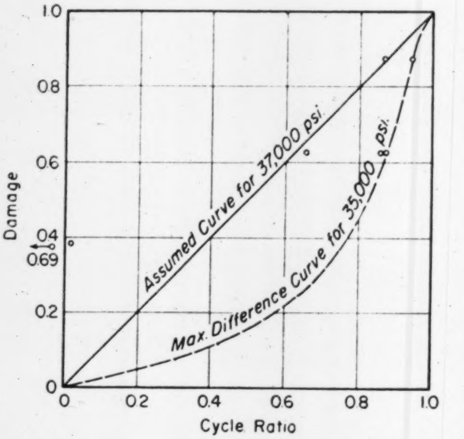


FIG. 19.—Results of Tests of Large Plate Specimens Using 37,000 Psi. as Prestress and 34,500 as Test Stress. (Using Cycle Ratio Values from Average *S-N* Curve.)

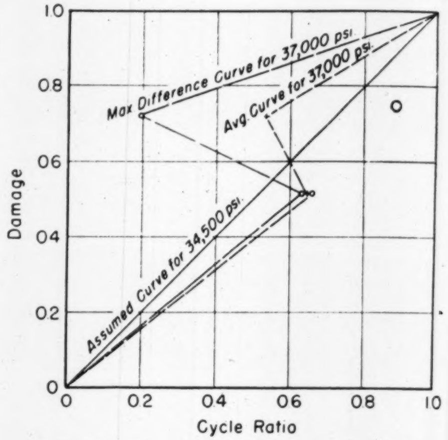


FIG. 20.—Results of Tests of Large Plate Specimens Using 34,500 psi. as Prestress and 37,000 psi. as Test Stress. (Using Values of Cycle Ratio from Average *S-N* Curve.)

TABLE VIII.—RESULTS OF TESTS TO DETERMINE CYCLE RATIO *VERSUS* DAMAGE CURVES FOR THE SMALL SPECIMENS OF A-7 STEEL.

Specimen No.	Prestress, psi.	Total Cycles Prestress	Test Stress, psi.	Total Cycles Test Stress	Results Based on Average Endurance Life			Results Based on Minimum Endurance Life[a]		
					C. R. P. S.	C. R. T. S.	Cum. C. R.	C. R. P. S.	C. R. T. S.	Cum. C. R.
32	53 000	80 000	45 000	27 000	0.690	0.027	0.717	1.212	0.047	1.259
33	53 000	60 000	45 000	341 000	0.518	0.341	0.859	0.909	0.600	1.509
34	53 000	80 000	45 000	155 000	0.690	0.155	0.845	1.212	0.272	1.484
35	53 000	40 000	45 000	482 000	0.345	0.482	0.827	0.606	0.846	1.452
36	53 000	20 000	45 000	607 000	0.173	0.607	0.780	0.303	1.066	1.369
37	53 000	60 000	45 000	289 000	0.518	0.289	0.807	0.909	0.508	1.417
38	53 000	20 000	45 000	732 000	0.173	0.732	0.905	0.303	1.284	1.587
39	53 000	80 000	45 000	543 000	0.690	0.543	1.233	1.212	0.952	2.164
40	53 000	80 000	45 000	125 000	0.690	0.125	0.815	1.212	0.219	1.431
42	53 000	60 000	45 000	276 000	0.518	0.276	0.794	0.909	0.485	1.394
43	53 000	40 000	45 000	631 000	0.345	0.631	0.976	0.606	1.107	1.713
44	53 000	40 000	45 000	349 000	0.345	0.349	0.694	0.606	0.612	1.218
53	53 000	40 000	45 000	342 000	0.345	0.342	0.687	0.606	0.601	1.207
54	53 000	20 000	45 000	601 000	0.173	0.601	0.774	0.303	1.055	1.358
45	45 000	200 000	53 000	149 000	0.200	1.280	1.480	0.351	2.260	2.611
46	45 000	200 000	53 000	107 000	0.200	0.923	1.123	0.351	1.620	1.971
47	45 000	766 000	Failed During Prestress		0.766	0	0.766	1.343	0	1.343
48	45 000	600 000	53 000	130 000	0.600	1.120	1.720	1.052	1.970	3.022
49	45 000	600 000	53 000	186 000	0.600	1.604	2.204	1.052	2.820	3.872
55	56 000	32 500	48 000	63 000	0.625	0.140	0.765	1.083	0.252	1.335
56	56 000	32 500	48 000	190 000	0.625	0.422	1.047	1.083	0.760	1.843
57	56 000	32 500	48 000	92 000	0.625	0.204	0.829	1.083	0.368	1.451
58	56 000	56 500	48 000	0	1.086	0	1.086	1.884	0	1.884
63	56 000	32 500	48 000	89 000	0.625	0.198	0.823	1.083	0.356	1.439
59	48 000	235 000	56 000	30 000	0.523	0.577	1.100	0.940	1.000	1.940
60	48 000	235 000	56 000	81 000	0.523	1.560	2.083	0.940	2.700	3.640
61	48 000	235 000	56 000	18 000	0.523	0.346	0.869	0.940	0.600	1.540
62	48 000	235 000	56 000	5 000	0.523	0.096	0.619	0.940	0.167	1.107
14	48 000	235 000	56 000	53 000	0.523	1.020	1.543	0.940	1.770	2.610

Average Endurance Life 45 000 psi. — 10^6; 48 000 psi. — 4.5×10^5; 53 000 psi. — 1.16×10^6; 56 000 psi. — 52,000
Minimum Endurance Life 45 000 psi. — 5.7×10^5 ;48 000 psi. — 2.5×10^5; 53 000 psi. — 66,000; 56 000 psi. — 30,000

[a] C. R. = cycle ratio. P. S. = prestress. T. S. = test stress. Cum. C. R. = cumulative cycle ratio.

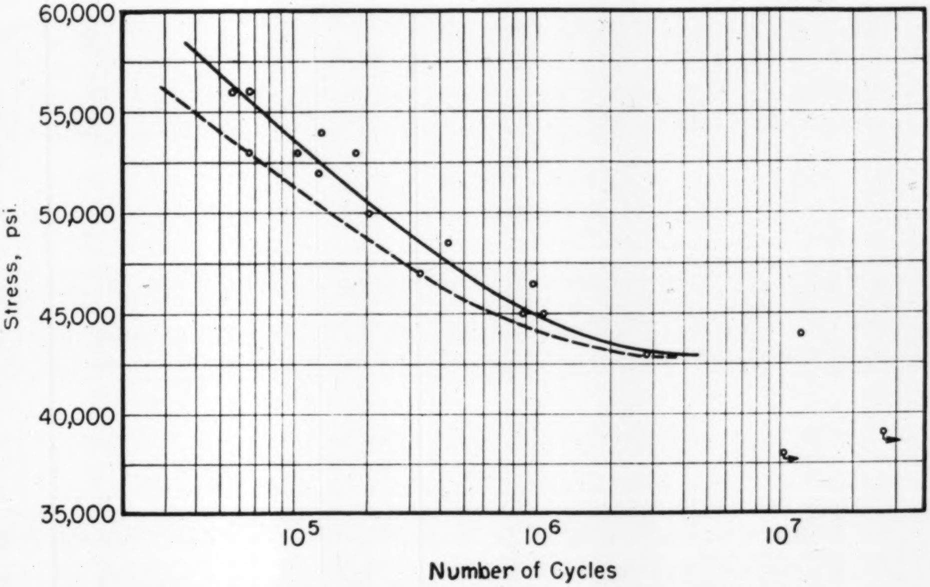


FIG. 21.—*S-N* Diagram for Small Rotating Beam. Specimens of A-7 Steel.

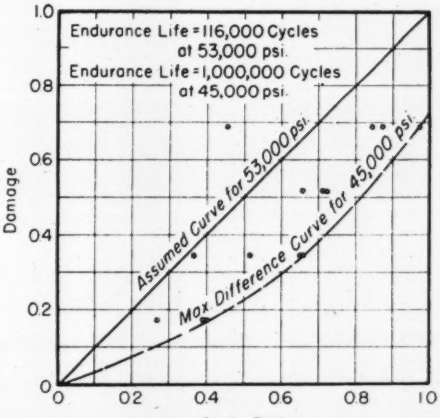


FIG. 22.—Results of Tests of Small Specimens of A-7 Steel Using 53,000 psi. as Prestress and 45,000 psi. as Test Stress.

than 1.0. As shown on Fig. 18 this value might be as high as 1.63 when the minimum *S-N* curve was used as the basis for computing endurance life and cycle ratio at the various stresses. The only way to eliminate the effect of the normal scatter involved when dealing with endurance life was to use the minimum values only. If we used values of endurance life and cycle ratio as determined from the minimum *S-N* curve, then we would expect to find all of the test and computed values of cumulative cycle ratio equal to or greater than 1.0 if the true damage *versus* cycle ratio relation were the same at all stress levels. However, Fig. 18 shows the test and computed results, based on the minimum *S-N* curve, of which several of the test points and the minimum computed curve lie below 1.0. This supports the assumption that a different damage-cycle ratio relation exists at different stress levels. Also Figs. 14 and 16 indicated that the higher prestress reduced the endurance life at the lower test stress considerably, but when a given cycle ratio of the lower stress was used as the prestress, it produced a smaller reduction of the endurance life at the higher stress. This corresponded with the results obtained by both Kommers (5) and Bennett (1). Figure 16 indicated that a prestress of about 0.43 cycle ratio at 32,000 psi. increased the endurance life at the test stress of 37,000 psi. to greater than

1.0. This increase might have been due to the prestress, but it might also have been due to the scatter of endurance life as shown on Fig. 13, or the limited number of tests run. The maximum difference curve shown on Fig. 16 represented the *least* effect on the endurance life at the test stress of 37,000 psi., produced by cycles of the prestress of 32,000 psi. If the *S-N* curve were actually a line instead of a band, the horizontal distances, or the cycle ratio relationships would be identical regardless of whether 37,000 psi. was used as the prestress and 32,000 psi. as the test stress, or *vice versa*. Then either Fig. 14 or Fig. 16 could be used for the subsequent computations of cumulative cycle ratio. However, since the *S-N* relationship for any given material is represented by this scatter band, it is most conservative to use values of cycle ratio based on the minimum *S-N* curve to determine the *D-R* curves. Then the curve of maximum difference on this *D-R* diagram which represents the *greatest* effect of the prestress upon the endurance life at the test stress should be used for computation of the value of cumulative cycle ratio with the stress-cycle pattern desired. Figures 14 and 15 illustrate how this was done for the tests of the large plate specimens using the stresses of 37,000 psi. and 32,000 psi.

TABLE IX.—RESULTS OF TESTS TO DETERMINE CYCLE RATIO *VERSUS* DAMAGE CURVES FOR THE S.A.E. 4340 STEEL SPECIMENS.[a]

Specimen No.	Prestress, psi.	Number Cycles At Prestress	Test Stress, psi.	Number Cycles At Test Stress	C. R. at P. S.	C. R. at T. S.	Cum. C. R.
M-1	130 000	28 000	105 000	17 000	0.800	0.100	0.900
R-1				66 000		0.388	1.188
S-4				97 000		0.571	1.371
M-2				4 000		0.024	0.824
U-4	130 000	17 500	105 000	68 000	0.500	0.400	0.900
T-4				205 000		1.260	1.760
P-1				111 000		0.653	1.153
D-2				62 000		0.365	0.865
K-4				116 000		0.682	1.182
M-4				99 000		0.582	1.082
Y-1				66 000		0.388	0.888
Y-3				201 000		1.182	1.682
M-3				46 000		0.271	0.771
A-1	130 000	17 500	105 000	100 000	0.500	0.588	1.088
A-4				34 000		0.200	0.700
H-2	130 000	7 000	105 000	352 000	0.200	2.071	2.271
V-2				142 000		0.835	1.035
H-3				156 000		0.918	1.118
D-1				127 000		0.747	0.947
Y-4				169 000		0.995	1.195
D-4				106 000		0.624	0.824
K-2				159 000		0.935	1.135
S-3				174 000		1.023	1.223
T-2				199 000		1.170	1.370
C-2				68 000		0.400	0.600
Z-2	105 000	136 000	130 000	33 000	0.800	0.943	1.743
L-1		136 000		3 000	0.800	0.086	0.886
Z-4		136 000		25 000	0.800	0.714	1.514
V-1		136 000		45 000	0.800	1.286	2.086
Y-2		68 000		40 000	0.400	1.144	1.544
F-1		68 000		35 000	0.400	1.000	1.400
E-4		68 000		36 000	0.400	1.028	1.428

[a] C. R. = cycle ratio. P. S. = prestress. T. S. = test stress. Cum. C. R. = Cumulative cycle ratio.

A series of tests was run using 37,000 psi. and 34,500 psi. for the prestress and test stresses. The results of these tests are shown on Figs. 19 and 20 and appear quite similar to the results from using 37,000 psi. and 32,000 psi. Only a few specimens were run to determine each curve, so they serve as illustrations of the procedure only. The alternate block tests to determine the value of cumulative cycle ratio were not run using these two stresses.

TABLE X.—RESULTS OF ALTERNATE BLOCK TESTS OF THE S.A.E. 4340 STEEL.

Specimen No.	Size Blocks	Stress for First Block, psi.	Total Cycles at 130,000 psi.	Total Cycles at 105,000 psi.	Total Cycle Ratios at 130,000 psi.	Total Cycle Ratios at 105,000 psi.	Value of Cumulative Cycle Ratio
G-4	0.30	130 000	21 000	91 000	0.600	0.535	1.135
W-2	0.30	130 000	28 000	102 000	0.800	0.600	1.400
K-1	0.30	130 000	21 000	83 000	0.600	0.488	1.088
F-2	0.30	130 000	21 000	79 000	0.600	0.465	1.065
L-3	0.30	105 000	21 000	128 000	0.600	0.753	1.353
N-4	0.30	105 000	21 000	109 000	0.600	0.641	1.241
G-1	0.30	105 000	28 000	153 000	0.800	0.900	1.700
B-2	0.20	130 000	18 000	68 000	0.514	0.400	0.914
K-3	0.20	130 000	14 000	60 000	0.400	0.353	0.753
G-2	0.20	130 000	20 500	68 000	0.585	0.400	0.985
G-3	0.20	130 000	15 000	68 000	0.428	0.400	0.828
R-4	0.20	130 000	13 000	34 000	0.371	0.200	0.571
J-2	0.20	130 000	28 000	123 000	0.800	0.723	1.523
D-3	0.20	105 000	14 000	84 000	0.400	0.494	0.894
J-1	0.20	105 000	24 500	136 000	0.700	0.800	1.500
C-3	0.20	105 000	14 000	99 000	0.400	0.583	0.983
J-3	0.10	130 000	24 500	111 000	0.700	0.653	1.353
J-4	0.10	130 000	14 000	53 000	0.400	0.312	0.712
B-1	0.10	130 000	18 000	88 000	0.514	0.518	1.032
W-4	0.10	105 000	29 000	153 000	0.828	0.900	1.728
L-4	0.10	105 000	33 500	136 000	0.958	0.800	1.758
H-1	0.10	105 000	24 500	119 000	0.700	0.700	1.400
Using $\frac{\text{Size of Cycle Ratio Block at 130 000 psi.}}{\text{Size of Cycle Ratio Block at 105 000 psi.}} = \frac{5}{1}$							
N-1	0.50/0.10	130 000	35 000	18 000	1.000	0.106	1.106
S-1	0.50/0.10	130 000	49 500	51 000	1.700	0.300	2.000
H-4	0.50/0.10	130 000	28 000	17 000	0.800	0.100	0.900
P-3	0.50/0.10	130 000	36 000	34 000	1.029	0.200	1.229
F-3	0.20/0.04	130 000	35 000	34 000	1.000	0.200	1.200
Q-4	0 20/0.04	130 000	29 000	28 000	0.829	0.165	0.994
W-2	0.20/0.04	130 000	37 000	35 000	1.057	0.206	1.263
Q-3	0.20/0.04	130 000	36 500	35 000	1.043	0.206	1.249

Tests of Small Rotating-Beam Specimens of A-7 Steel:

The tests to determine the *S-N* curve and the prestress - test stress type tests were run on small rotating-beam specimens of this A-7 steel also. The results of these tests are given in Table VIII and are shown in Figs. 21 and 22. Figure 21 shows the *S-N* diagram for these small specimens. As in the large specimen tests, the nominal stress was used rather than to attempt to determine the stress distribution across the test section after stressing some fibers above the yield point stress. The yield point of this steel was about 39,000 psi., thus for all the tests shown on Fig. 21 the extreme fibers of the test section had been subjected to some inelastic action.

The *D-R* curves obtained from these small specimens were very similar in appearance to those obtained for the plate specimens. These tests of small specimens of the A-7 steel were performed to determine whether the scatter of test points would be so great that this method of determining the value of cumulative cycle ratio from the *D-R* curves would be useless or not. Figure 22 showed that, although the scatter of test points was considerable, the maxi-

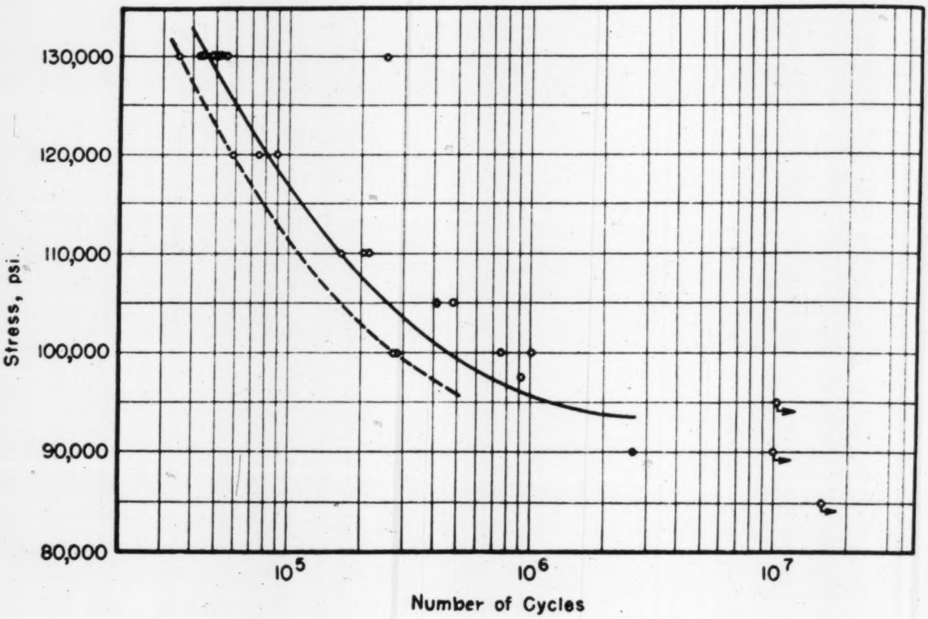


FIG. 23.—*S-N* Diagram for Small Specimens of S.A.E. 4340 Steel.

mum difference curve could be established experimentally for subsequent use in computations.

Tests of Small Rotating Beam Specimens of S.A.E. 4340 Steel:

Since the tests of the small A-7 steel specimens indicated that the results obtained, using the small specimens in the high-speed fatigue machines, were consistent enough to produce smooth *D-R* envelope curves, a further set of tests was run on small specimens of S.A.E. 4340 steel. These results are given in Tables IX and X, and on Figs. 23, 24, 25, and 26. This S.A.E. 4340 steel was supposed to be of aircraft quality, but proved to be very inhomogeneous due to "banding" in the center of the 3-in. diameter bar from which these small specimens were taken. Thus the *S-N* diagram and subsequent figures showed excessive scatter, and it was necessary to run numerous tests for each condition to be sure minimum values were obtained. The *S-N* diagram shown on Fig. 23 gives the clearest indication of the scatter involved. At the stress of 130,000 psi., the maximum endurance life was found to be 258,000 cycles and the minimum, 35,000 cycles. The speci-

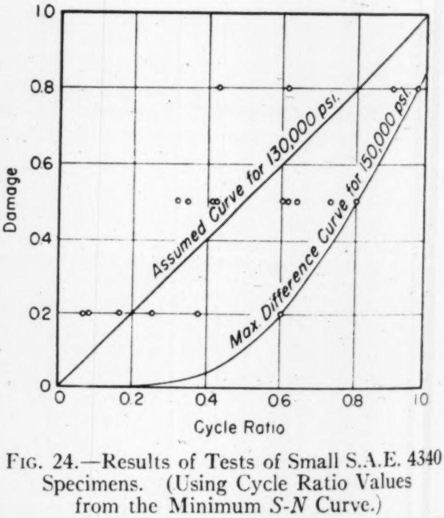


FIG. 24.—Results of Tests of Small S.A.E. 4340 Specimens. (Using Cycle Ratio Values from the Minimum *S-N* Curve.)

men having the maximum endurance life had been taken from near the surface of the 3-in. diameter parent bar while the specimen showing the minimum came from near the center. An attempt was made in all tests to use specimens from different positions in the parent bar in each test series to provide typical results. However, practically all the minimum curve values were from tests of specimens near the center of the parent bar.

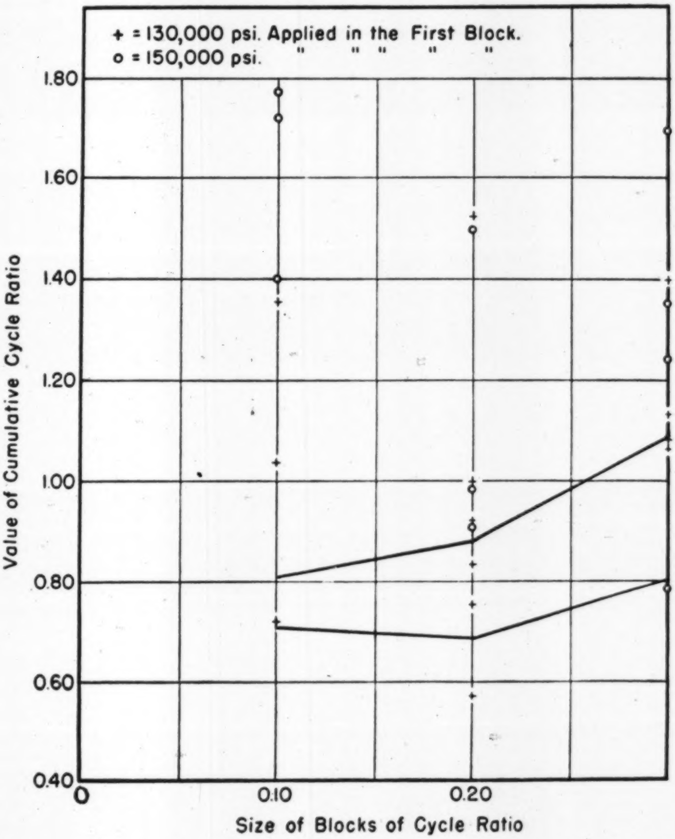


FIG. 25.—Effect of Sizes of Blocks of Cycle Ratio Used S.A.E. upon Value of Cumulative Cycle Ratio for Small Specimens of 4340 Steel. (Using Cycle Ratio Values from the Minimum S-N Curve.)

Figure 25 shows the comparison between the test results for the alternate block tests and the minimum curve as computed from the experimentally determined *D-R* curves. Only one test point fell below this minimum curve and probably if more tests had been run to determine the *D-R* curves, this computed curve could have been lowered to include this stray test point. Similarly on Fig. 26 where a 5 to 1 ratio of the sizes of blocks of cycle ratio at 37,000 psi. to those at 32,000 psi. was used, one test point fell slightly below the minimum curve. However, in this case, the discrepancy was almost negligible. Another factor which tended to reduce the value of cumulative cycle ratio obtained in these alternate block type tests was the number of times the machines were stopped and started during the test. In the alternate block tests, the machine was stopped from 3 to 17 times depending upon the size of the blocks of cycle ratio used and the endurance of the specimen. A few check tests at a constant stress to determine the effects of numerous stops on the test results showed no noticeable effects of the stops and starts. However, the test section of these specimens was only 0.105 in. in diameter, consequently it was possible to bend them unless extreme care was taken.

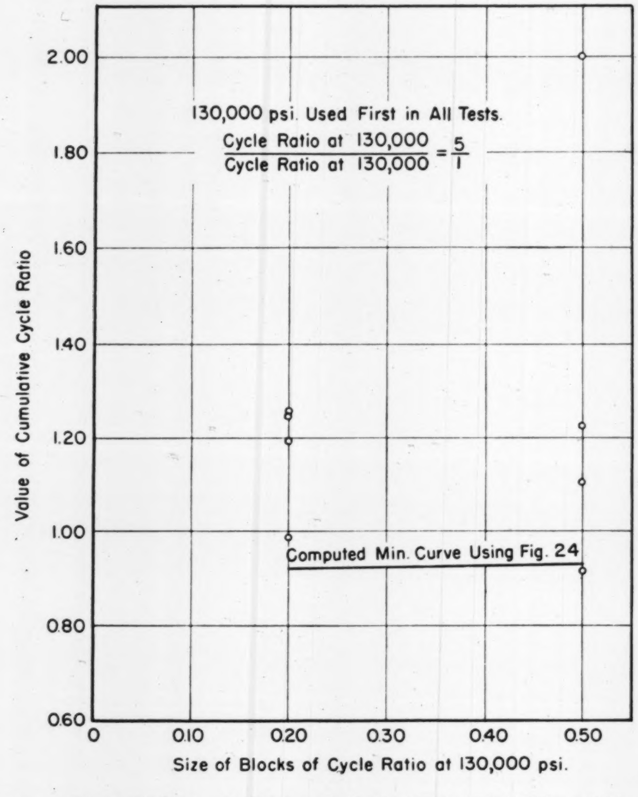


FIG. 26.—Effect of Unequal Sizes of Blocks of Cycle Ratio Used Upon Value of Cumulative Cycle Ratio for Small Specimens of S.A.E. 4340 Steel.

CONCLUSIONS

This investigation indicated that it is reasonable to assume that a curve exists on the damage *versus* cycle ratio diagram which represents the progress of failure at a given stress. This curve is continuous and is different when it represents different levels of stress. These curves may be used in groups of two or more to determine the value of cumulative cycle ratio available when various amounts of cycle ratio are applied at each stress. The value of cumulative cycle ratio is a number which represents the life of the specimen, and is a function of the total number of cycles available at the different stresses before failure.

When two curves are used and small

equal values of cycle ratio are considered to be applied alternately to failure, the theoretical value of cumulative cycle ratio obtained is always less than 1.0. Greater divergence of the two curves produces a larger reduction of this value from 1.0. When large equal values of cycle ratio are applied, the order in which the curves are used produces a significant effect on the value of cumulative cycle ratio.

The theoretical value of cumulative cycle ratio is found to depend on the relative position of the curves on the *D-R* diagram rather than upon their exact position. The relative positions of the curves must be such that the proper relation is maintained between the values of cycle ratio at the various stress levels involved. Using this fact that relative curves are sufficient for further computations, a method is indicated whereby these relative *D-R* curves can be determined experimentally. After these experimentally determined curves are obtained, they can be used for calculation of the value of cumulative cycle ratio for any stress-cycle pattern desired.

The results of calculations, based on the hypothesis, for multiple stress level tests shows that for small alternately applied blocks of cycle ratio the value of cumulative cycle ratio is always less than 1.0. This is true even if all values of cycle ratio are based on the minimum *S-N* curve. Thus a procedure such as Miner's (6), which predicts failure at a value of cumulative cycle ratio equal to 1.0, errs on the unsafe side. That is, actual failure of the material might occur before it was supposed to, according to the prediction by such a procedure.

The experimental results indicated that considerable scatter was encountered in the determination of the *D-R* curves. However, a minimum curve, or maximum difference curve, could be determined which would give minimum values for all further computations. This was desirable since designs are usually based on minimum values. The test results for the large plate specimens agreed well with the minimum curve on the value of cumulative cycle ratio *versus* size of block of cycle ratio diagram. This minimum curve was obtained from computations based on the assumed curve and the maximum difference curve from the *D-R* diagram. The test results for the small specimens of S.A.E. 4340 steel showed more than the usual scatter. This was probably due to the inhomogeneity of the grain structure, but in spite of this excess scatter, all but one of the test values fell above the computed minimum values of cumulative cycle ratio.

It appears that the procedure described in this paper for combining the cumulative damaging effects, produced by various stress levels, to give an estimate of the life of the material, is workable. However, the relative *D-R* curves must be determined experimentally as part of the procedure and hence this method cannot be used unless it is possible to run a number of tests to determine these *D-R* curves.

REFERENCES

(1) J. A. Bennett, "A Study of the Damaging Effect of Fatigue Stressing on X4130 Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 693 (1946).

(2) H. J. French, "Fatigue and Hardening of Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, pp. 899–946 (1933).

(3) J. B. Kommers, "The Effect of Understressing on Cast Iron and Open Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, p. 368 (1930).

(4) J. B. Kommers, "The Effect of Overstressing and Understressing in Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 249 (1938).

(5) J. B. Kommers, "The Effect of Overstress in Fatigue on the Endurance Life of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 532 (1945).

(6) M. A. Miner, "Cumulative Damage in Fatigue," *Journal of Applied Mechanics*, Vol. 12, September, 1945, p. A-159.

DISCUSSION

Mr. John M. Lessells.[1]—In Miner's original work, which the author has now considerably extended, a large "scatter" in results was obtained and this was taken into account in the analysis of results. Did the present authors do likewise?

A study of Miner's[2] work seems to indicate that when the stress is reduced in a series of stages, each level being in excess of the endurance limit, the value of $\sum \frac{n}{N}$ is less than unity whereas when the stress is increased in a series of stages the above value is greater than unity. Tests made at the Massachusetts Institute of Technology on steels seem to give confirmation. The views of the authors on this point would be of interest.

Mr. B. F. Langer[3] (*by letter*).—The work described in this paper materially increases the practical usefulness of the facts already known about the fatigue of metals. The designer of machinery needs not only the data from fatigue tests, but also work of this type to help him use the data intelligently.

All designers, from time to time, take cognizance of the fact that stresses which occur infrequently can be allowed to rise to higher values than those which occur many millions of times during the life of a machine. Members with steady load are designed on the basis of the yield strength, whereas members with vibratory load are designed on the basis of the endurance limit. When the amplitude of vibration fluctuates, there is no adequate method for predicting the life of the member. An important example is the railroad axle. Bending stress in each fiber fluctuates through a full cycle each wheel revolution. Due to track irregularities, very high loads can occasionally occur, but the highest values may come only a few thousand times during the life of the axle. Lower values occur with greater frequency, and the over-all duty of the axle may be represented by a series of points showing frequency per mile *versus* stress level. (The plot should not be drawn as a continuous curve, since the total number of cycles per mile at all stress levels should be finite and should not exceed the number of wheel revolutions.)

It is not practical to design railroad axles so that the highest stress which will ever be applied is lower than the endurance limit. It appears that the application of the authors' method to the stress-frequency data for an axle would be very laborious. It is to be hoped that some more simple approximation can be devised, perhaps involving a fractional factor, depending on the material, to represent the cycle ratio at which failure occurs. It should be remembered that a liberal amount of approximation is tolerable, since the scatter which occurs in all fatigue tests, particularly in the upper branch of the S-N curve, lowers the accuracy and reliability of any formula and necessitates the use of a liberal safety factor.

---

[1] Associate Professor of Mechanical Engineering, Engineering, Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[2] M. A. Miner, "Cumulative Damage in Fatigue," *Journal of Applied Mechanics*, Vol. 12, September, 1945, p. A-159

[3] Manager, Mechanical Section, Transportation Engineering Dept., Westinghouse Electric Corp., East Pittsburgh, Pa.

Most practical applications, such as the railroad axle, involve the application of very small blocks of cycle ratio. Therefore, the writer suggests that future work be concentrated on the study of the effects of many different stresses applied in small blocks. Test data from this type of loading can be obtained on standard fatigue machines by loading through springs instead of weights and then varying the spring force with a motor-driven cam.

MESSRS. F. E. RICHART, JR. AND N. M. NEWMARK (*authors' closure*).—The authors appreciate the comments by Mr. Lessells and Mr. Langer.

The scatter in the test results was considered in the interpretation of the results. Because of the scatter emphasis was placed on minimum curves rather than average values in drawing conclusions from the test results.

Regarding Mr. Lessells' observation that the cumulative cycle ratio can be greater than unity when the stresses are increased in small stages, this is known to be true and has been demonstrated by various tests. However this observation does not appear to be valid when the stresses are increased by large increments, nor does it seem valid for random applications of stresses of varying intensities. For such conditions it appears that a cumulative cycle ratio of less than one could be used in design. However when service conditions similar to the conditions required for "coaxing" can be counted on, some allowance for this can be made in the design.

In answer to Mr. Langer's comment, possibly a suitable design procedure would be to assume that failure can occur at some arbitrary cumulative cycle ratio, say perhaps 0.80, when entirely random stress frequencies can be encountered.

Further studies of these problems are contemplated and it is hoped that the additional data that Mr. Langer suggests are neccessary will eventually be available.

THE EFFECT OF SMALL PERCENTAGES OF SILVER AND COPPER ON THE CREEP CHARACTERISTICS OF EXTRUDED LEAD*

By G. R. Gohn[1] and W. C. Ellis[1]

Synopsis

Creep tests on longitudal tension test specimens taken from a series of lead-silver alloys extruded in the form of 0.140 in. thick pipe and containing various percentages of copper indicate that silver up to 0.010 per cent improves the creep resistance at stresses of 400 psi. and above. The improvement at 400 psi. was only about one fourth that observed at higher stresses. At 250 psi. no appreciable improvement was observed, while at lower stresses the effect of silver was indeterminate after 16,000 hr. However, the convergence of the creep rate—stress curves at low stress indicates that the effect of silver at stresses below 250 psi. is small. No appreciable improvement in creep rate resulted from an increase in silver content beyond 0.010 per cent or from the addition of 0.061 per cent of copper to the higher silver alloy.

It has been recognized for many years that the elements commonly present as impurities or as intentional small additions in lead are important in determining creep behavior. However, the specific effects of many individual elements alone or in controlled combinations have not been clearly delineated. The present paper deals with the effect of combinations of silver and copper on the creep characteristics of lead extruded in the form of 0.140-in. thick, water-quenched pipe samples containing approximately 0.005 per cent bismuth and 0.002 per cent sodium as the only other constituents present in appreciable quantities. A companion paper by Phelps, Kahn and Magee **(1)**[2] of the Philadelphia Electric Co. correlates the creep data obtained from these tests on flattened tensile specimens with bursting tests made by them on pipe samples of the same series of alloys.

Review of Previous Work

The literature contains many reports on the creep resistance of silver- and copper-bearing leads. The effect of both silver and copper has been carefully investigated by Greenwood and his coworkers and is reported in a series of papers **(2) (3) (4)**. However, their conclusions are based almost entirely on tests made with specimens from cast-and-rolled or from cast, rolled, and annealed material. In electrolytic lead having total impurities of 0.0005 per cent Russell **(2)**, a coworker of Greenwood, found that at stresses of 500 psi. and with silver contents of 0.0001 to 0.005 per cent, the creep rate decreased with an increase in silver content. At a lower stress of 350 psi. the presence of silver in the lead had a less marked effect on the creep resistance. Greenwood and Worner **(3)** using a less pure base lead containing 0.002 per cent bis-

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 814.

TABLE I.—CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF LEAD AND LEAD-ALLOY PIPE SAMPLES.

Alloy	Composition, per cent[a]					Tensile Strength, psi.	Elongation in 2 in., per cent
	Silver	Copper	Bismuth	Sodium	Lead[b]		
No. 30	0	0	0.004	0.001	99.995	1740	67.5
No. 31	0.001	0.004	0.005	0.002	99.988	1860	63.5
No. 32	0.003	0.004	0.005	0.002	99.986	1940	55.5
No. 33	0.010	0.004	0.005	0.002	99.979	2060	49.0
No. 34	0.018	0.004	0.005	0.002	99.971	2220	41.0
No. 35	0.017	0.061	0.005	0.002	99.915	2320	46.0

[a] Analyses supplied by the Okonite-Callendar Cable Co., Inc.
[b] Lead by difference.

TABLE II.—CREEP CHARACTERISTICS OF LONGITUDINAL TEST SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 30—0 per cent silver, 0 per cent copper. Tested at 85 F., 3-in. gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
1500	1	0	0.0253	....	....	....	....	
		0.9 (F)	0.512[a]	5690	5400	....	....	Ductile
1000	1	0	0.0027	....	....	....	....	
		25	0.0572	22.9	21.8	11.7	0.0280	
		78.5 (T)	0.1200	15.3	11.7	11.7 (*v*)	0.0280 (ϵ_0)	
		100	0.1685	16.8	22.5	16.1	....	
		143.1 (F)	0.489[a]	34.2	74.5	....	....	Ductile
800	1	0	0.	....	....	....	....	
		100	0.0445	4.45	4.45	3.65	0.0080	
		250	0.0893	3.58	3.00	2.37	0.0300	
		570 (T)	0.1650	2.89	2.37	2.37 (*v*)	0.0300 (ϵ_0)	
		750	0.2690	3.59	5.78	20.6	....	
		836 (F)	0.436[a]	5.22	19.4	....	....	Ductile
600	1	0	0.	....	....	....	....	
		100	0.0044	0.44	0.44	0.29	0.0015	
		1 000	0.270	0.27	0.26	0.26	0.0015	
		2 700 (T)	0.0717	0.26	0.26	0.26 (*v*)	0.0015 (ϵ_0)	
		3 000	0.0805	0.27	0.29	0.31	....	
		6 197 (F)	0.308[a]	0.50	0.71	....	....	Brittle
400	2	0	0.	....	....	....	....	
		1 000	0.0020	0.020	0.020	0.018	0.0002	
		2 000	0.0033	0.017	0.013	0.015	0.0003	
		5 000	0.0076	0.015	0.014	0.012	0.0017	
		10 000	0.0132	0.013	0.011	0.012	0.0017	
		16 000	0.0203	0.012	0.012	0.012 (*v*)	0.0017 (ϵ_0)	No failure
250	2	0	0.	....	....	....	....	
		1 000	0.0007	0.0070	0.0070	0.0023	0.0005	
		2 000	0.0010	0.0050	0.0030	0.0023	0.0005	
		5 000	0.0016	0.0032	0.0020	0.0023	0.0005	
		10 000	0.0028	0.0028	0.0024	0.0023	0.0005	
		16 000	0.0042	0.0027	0.0023	0.0023 (*v*)	0.0005 (ϵ_0)	No failure
150	2	0	0.	....	....	....	....	
		16 000	0.0007	0.00044	....	<0.00044[c] (*v*)	....	No failure
100	2	0	0.	....	....	....	....	
		16 000	0.0004[b]	0.00022	....	<0.00044[c](*v*)	....	No failure

[a] Elongation after failure.
[b] The least reading which can be measured with the cathetometer is 0.0007 in. per inch on a 3-in. gage length.
[c] Value obtained by dividing least reading by elapsed time.

NOTES:

(F) Denotes failure of test specimen after time interval indicated.
(T) Indicates time at which third stage of creep begins.
(*v*) Minimum tangent creep rate observed during test period.
(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

muth and 0.004 per cent antimony added 0.01, 0.05, and 0.1 per cent of silver or copper. Curves based upon these tests indicate that at a stress of 500 psi., both silver and copper diminish the creep rate of lead. On the other hand, at a stress of 350 psi. the creep rate was increased by the presence of the silver and the copper except for the alloy containing 0.1 per cent of copper for which alloy the creep rate was substantially reduced. Greenwood and Cole (4) in 1947 summarized the findings of this group of workers by stating that silver in amounts up to 0.1 per cent does not greatly influence the creep rate of lead while copper in amounts over 0.05 per cent reduces the creep rate by a factor of 25.

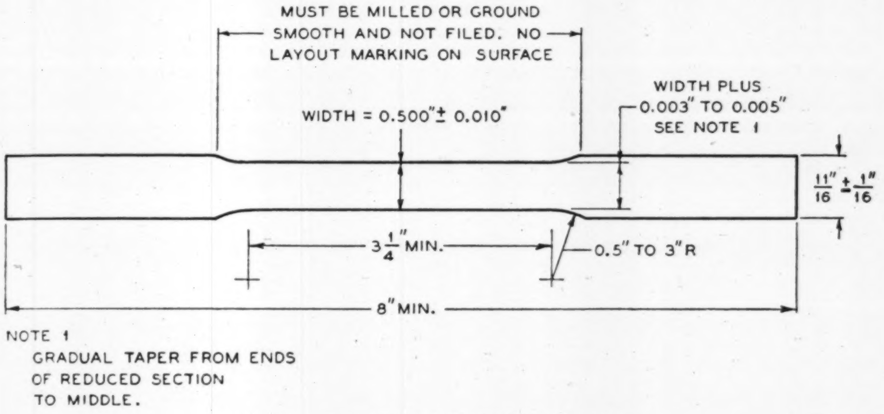

MUST BE MILLED OR GROUND SMOOTH AND NOT FILED. NO LAYOUT MARKING ON SURFACE
WIDTH = 0.500"± 0.010"
WIDTH PLUS 0.003" TO 0.005" SEE NOTE 1
11"/16 ± 1"/16
3 1/4" MIN.
0.5" TO 3" R
8" MIN.
NOTE 1
GRADUAL TAPER FROM ENDS OF REDUCED SECTION TO MIDDLE.

FIG. 1.—Tension and Creep Test Specimen.

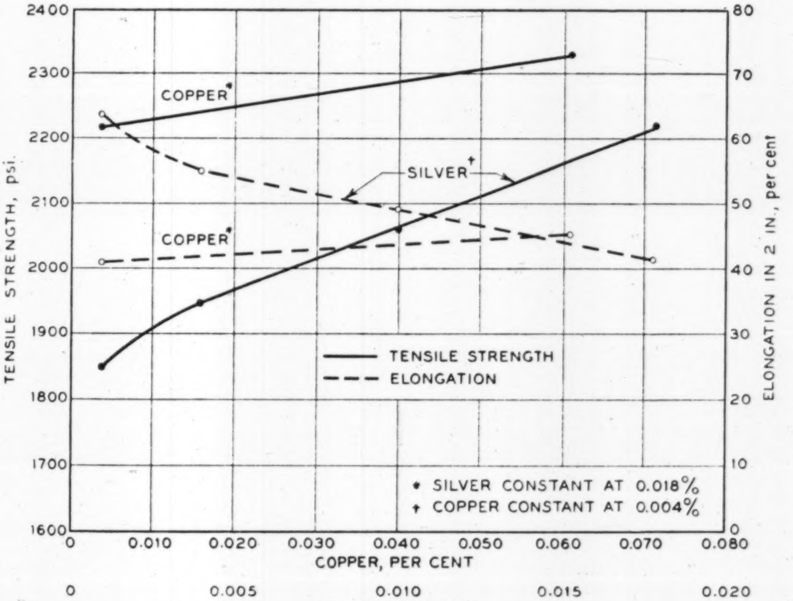


FIG. 2.—Effect of Copper and Silver on Tensile Strength of Lead Cable Sheathing Alloys.

A. J. Phillips (5), also using cast and rolled specimens, has reported that the addition of copper to lead improves the creep resistance. On extruded tape samples Gohn, Arnold, and Bouton (6) found, in tests made at 85 F. at stresses of 150 to 250 psi. extending to 70,000 hr. that chemical lead containing 0.05 to 0.06 per cent copper and approximately 0.005 per cent silver was superior in creep resistance to high-purity lead. Creep rates given for lead and its alloys in the joint A.S.T.M.-A.S.M.E. 1938 compilation of creep data (7) show at stresses of 150 to 250 psi. and a test temperature of 110 F. a contrary conclusion in that chemical lead had approximately twice the creep rate of corroding lead. However, these tests were only of 2000 to 10,000 hr. duration and, unfortunately, the type and treatment of the specimens were not included.

TABLE III.—CREEP CHARACTERISTICS OF LONGITUDINAL SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 31—0.001 per cent silver, 0.004 per cent copper. Tested at 85 F., 3-in. gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
1500......	1	0	0.053	....	....	....	....	
		3.9 (F)	0.483[a]	1240	1100	....	....	Ductile
1000......	1	0	0.0027	....	....	....	....	
		25	0.0341	13.6	12.6	8.84	0.0120	
		50	0.0546	10.9	8.02	6.93	0.0200	
		182 (T)	0.1460	8.03	6.93	6.93 (v)	0.0200 (ϵ_0)	
		250	0.3150	12.6	7.37	12.6	....	
		271 (F)	0.474[a]	17.5	7.57	....	....	Ductile
800......	1	0	0.	....	....	....	....	
		100	0.0204	2.04	2.04	1.79	0.0025	
		250	0.0453	1.81	1.66	1.37	0.0110	
		872 (T)	0.1300	1.49	1.37	1.37 (v)	0.0110 (ϵ_0)	
		1 000	0.1580	1.58	2.19	2.64	....	
		1 330 (F)	0.426[a]	3.20	8.13	....	....	Ductile
600......	1	0	0.	....	....	....	....	
		100	0.0007	0.070	0.070	0.074	0.	
		1 990 (T)	0.0147	0.074	0.074	0.074 (v)	0. (ϵ_0)	
		2 500	0.0200	0.080	0.10	0.13	....	
		12 163 (F)	0.430 (F)	0.35	0.42	....	....	Ductile
400......	2	0	0.	....	....	....	....	
		1 000	0.0014	0.014	0.014	0.014	0.	
		2 000	0.0028	0.014	0.014	0.014	0.	
		5 000	0.0070	0.014	0.014	0.014	0.	
		10 000 (T)	0.0140	0.014	0.014	0.014 (v)	0. (ϵ_0)	
		16 000	0.0241	0.015	0.017	0.024	....	No failure
250......	2	0	0.	....	....	....	....	
		1 000	0.0006	0.0060	0.0060	0.0025	0.0003	
		2 000	0.0008	0.0040	0.0040	0.0025	0.0003	
		5 000	0.0016	0.0032	0.0027	0.0025	0.0003	
		10 000	0.0028	0.0028	0.0024	0.0025	0.0003	
		16 000	0.0043	0.0027	0.0025	0.0025 (v)	0.0003 (ϵ_0)	No failure
150......	2	0	0.	0.	....	....	....	
		16 000	0.0029	0.0018	0.0018	<0.0018[b] (v)	0. (ϵ_0)	No failure
100......	2	0	0.	0.	....	....	....	
		16 000	0.0013	0.00080	0.00080	<0.00080[b] (v)	0. (ϵ_0)	No failure

[a] Elongation after failure.
[b] Value obtained by dividing total creep by elapsed time.

NOTES:
(F) Denotes failure of test specimen after time interval indicated.
(T) Indicates time at which third stage of creep begins.
(v) Minimum tangent creep rate observed during test period.
(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

The findings of the various investigators are not in complete accord. This is not unexpected in so complex a process as creep in which a number of factors in addition to composition are important in determining creep behavior. For example, it is well known that the metallurgical process used in preparing the specimens and the heat treatment as it affects the internal structure, are both important factors in determining creep resistance. In some instances the preparation may even overshadow the effect of composition. The data in the literature also indicate (in some instances) that the comparative creep resistance of two lead alloys may be reversed with a change in stress level. This appears to have occurred in the lead-silver alloys discussed by Greenwood and Worner (3) and has also been reported by Gohn,

TABLE IV.—CREEP CHARACTERISTICS OF LONGITUDINAL SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 32—0.003 per cent silver, 0.004 per cent copper. Tested at 85 F., 3-in. gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
1500......	1	0	0.0067	....	....	....	....	Ductile
		10.5 (F)	0.468[a]	446.	438.	....	....	
1000......	1	0	0.	....	....	....	....	
		50.	0.0120	2.40	2.40	2.19	0.0010	
		360 (T)	0.0800	2.22	2.19	2.19 (v)	0.0010 (ϵ_0)	
		500	0.1246	2.49	3.20	3.83	....	
		815 (F)	0.473[a]	5.81	11.1	....	....	Ductile
800......	1	0	0.0007	....	....	....	....	
		100	0.0080	0.80	0.73	0.71	0.0010	
		750 (T)	0.0540	0.72	0.71	0.71 (v)	0.0010 (ϵ_0)	
		1 000	0.0820	0.82	1.12	1.06	....	
		2 535 (F)	0.574[a]	2.26	3.21	....	....	Ductile
600......	1	0	0.	....	....	....	....	
		100	0.0004	0.040	0.040	0.040	0.	
		1 000	0.0035	0.035	0.034	0.034	0.	
		10 400 (T)	0.0360	0.035	0.034	0.034 (v)	0. (ϵ_0)	
		12 000	0.0445	0.037	0.053	0.067	....	
		16 000	0.0830	0.052	0.096	0.13	....	No failure
400......	2	0	0.	....	....	....	....	
		1 000	0.0010	0.010	0.010	0.0097	0.	
		2 000	0.0020	0.010	0.010	0.0097	0.	
		5 000	0.0049	0.0098	0.010	0.0097	0.	
		10 000	0.0098	0.0098	0.0098	0.0097	0.	
		16 000	0.0155	0.0097	0.0097	0.0097 (v)	0. (ϵ_0)	No failure
250......	2	0	0.	....	....	....	....	
		1 000	0.0004[b]	0.0040	0.0040	0.0042	0.	
		2 000	0.0008	0.0040	0.0040	0.0034	0.0001	
		5 000	0.0018	0.0036	0.0033	0.0018	0.0009	
		10 000	0.0027	0.0027	0.0018	0.0018	0.0009	
		16 000	0.0038	0.0024	0.0018	0.0018 (v)	0.0009 (ϵ_0)	No failure
150......	2	0	0.	....	....	....	....	
		16 000	0.0010	....	....	<0.00067[d] (v)	....	No failure
100......	2	0	0.	....	....	....	....	
		16 000	0.	....	....	<0.00044[c] (v)	....	No failure

[a] Elongation after failure.
[b] The least reading which can be measured with the cathetometer is 0.0007 in. per inch on a 3-in. gage length.
Value obtained by dividing least reading by elapsed time.
[d] Value obtained by dividing total creep by elapsed time.

NOTES:
(F) Denotes failure of test specimen after time interval indicated.
(T) Indicates time at which third stage of creep begins
(v) Minimum tangent creep rate observed during test period.
(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

Arnold, and Bouton (6) in the case of the lead-antimony and lead-tin alloys when compared with the chemical lead specimens.

TABLE V.—CREEP CHARACTERISTICS OF LONGITUDINAL SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 33—0.010 per cent silver, 0.004 per cent copper. Tested at 85 F., 3-in. gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
2000......	1	0	0.0240	....	....	....	....	Ductile
		0.42 (F)	0.427[a]	1020	960	....	....	
1500......	1	0	0.0060	....	....	....	....	
		5.5	0.0690	126	115	....	....	
		23.6 (F)	0.555[a]	235	206	....	....	Ductile
1000......	1	0	0.0013	....	....	....	....	
		50	0.0090	1.80	1.54	1.65	0.0010	
		540 (T)	0.0900	1.67	1.65	1.65 (*v*)	0.0010 (ϵ_0)	
		600	0.1075	1.79	2.92	1.80	....	
		1 244 (F)	0.628[a]	5.05	8.10	....	....	Ductile
800......	1	0	0.	....	....	....	....	
		100	0.0030	0.30	0.30	0.24	0.0004	
		820 (T)	0.0200	0.24	0.24	0.24 (*v*)	0.0004 (ϵ_0)	
		1 000	0.0285	0.28	0.47	0.32	....	
		4 352 (F)	0.560[a]	1.29	1.58	....	....	Ductile
600....	1	0	....			....	....	
		100	0.0003	0.030	0.030	0.030	0.	
		1 000	0.0026	0.026	0.026	0.026	0.	
		5 000 (T)	0.0130	0.026	0.026	0.026 (*v*)	0. (ϵ_0)	
		6 000	0.0160	0.027	0.030	0.031	....	
		10 000	0.0333	0.033	0.043	0.050	....	
		16 000	0.0675	0.043	0.057	0.058	....	No failure
400...	2	0	0.		....	....	....	
		5 000	0.0040	0.0080	0.0060	0.0050	0.0015	
		10 000	0.0065	0.0065	0.0050	0.0050	0.0015	
		16 000	0.0095	0.0059	0.0050	0.0050 (*v*)	0.0015 (ϵ_0)	No failure
250...	2	0	0.0004[b]			..	....	
		1 000	0.0007	0.0070	0.0030	0.0070	0.0000	
		2 000	0.0013	0.0065	0.0060	0.0060	0.0001	
		5 000	0.0024	0.0048	0.0037	0.0019	0.0014	
		10 000	0.0033	0.0033	0.0018	0.0019	0.0014	
		16 000	0.0045	0.0028	0.0020	0.0019 (*v*)	0.0014 (ϵ_0)	No failure
150.....	2	0	0.0004[b]	....	....	....	....	
		16 000	0.0010	0.00067	0.00038	<0.00067[d] (*v*)	....	No failure
100......	2	0	0.	....	....		....	
		16 000	0.[b]	....	....	<0.00044[c] (*v*)	....	No failure

[a] Elongation after failure.
[b] The least reading which can be measured with the cathetometer is 0.0007 in. per inch on a 3-in. gage length.
[c] Value obtained by dividing least reading by elapsed time.
[d] Value obtained by dividing total creep by elapsed time.

NOTES:
(F) Denotes failure of test specimen after time interval indicated.
(T) Indicates time at which third stage of creep begins.
(*v*) Minimum tangent creep rate observed during test period.
(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

MATERIALS INVESTIGATED

The alloys studied in the present investigation consisted of six compositions extruded in the form of pipe samples by the Okonite-Callendar Cable Co., Inc. primarily for bursting studies by the Philadelphia Electric Co. Through the kindness of Mr. H. S. Phelps of the latter company, sufficient material was made available to the authors for long-time tension (creep) and reversed-bending fatigue studies. Details concerning the extrusion of these alloys are given in the

TABLE VI.—CREEP CHARACTERISTICS OF LONGITUDINAL SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 34—0.018 per cent silver, 0.004 per cent copper. Tested at 85 F., 3-in. gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
2000......	1	0	0.0267	....	....	....	....	
		0.6 (F)	0.468[a]	7800	7340	....	....	Ductile
1500......	1	0	0.0600	....	....	....	....	
		25	0.1600	64	40	....	....	
		47.5 (F)	0.530[a]	111	164	....	....	Ductile
1000......	1	0	0.0007	....	....	....	....	
		25	0.0190	1.68	1.40	1.64	0.0150	
		360 (T)	0.0740	2.05	1.64	1.64 (v)	0.0150 (ϵ_0)	
		500	0.1125	2.25	2.75	2.97	....	
		815 (F)	0.387[a]	4.75	8.72	....	....	Brittle
800......	1	0	0.0007	....	....	....	....	
		100	0.0020	0.20	0.13	0.12	0.0007	
		1 000	0.0122	0.12	0.11	0.12	0.0007	
		1 020 (T)	0.0125	0.12	0.15	0.12 (v)	0.0007 (ϵ_0)	
		1 250	0.0195	0.16	0.30	0.29	....	
		2 796 (F)	0.191[a]	0.67	1.11	....	....	Brittle
600......	1	0	0.	....	....	....	....	
		1 000	0.0026	0.026	0.026	0.021	0.0005	
		2 000	0.0046	0.023	0.020	0.021	0.0005	
		5 000	0.0110	0.022	0.021	0.021	0.0005	
		6 800 (T)	0.0145	0.021	0.019	0.021 (v)	0.0005 (ϵ_0)	
		7 000	0.0152	0.022	0.035	0.029	....	
		10 000	0.0325	0.032	0.058	0.088	....	Brittle
		12 428 (F)	0.191[a]	0.15	0.65	....	....	
400......	2	0	0.	....	....	....	....	
		2 000	0.0013	0.0065	0.0065	0.0066	0.	
		5 000	0.0033	0.0066	0.0066	0.0066	0.	
		10 000	0.0066	0.0066	0.0066	0.0066	0.	
		16 000	0.0106	0.0066	0.0067	0.0066 (v)	0. (ϵ_0)	No failure
250......	2	0	0.				....	
		5 000	0.0011	0.0022	0.0022	0.0022	0.	
		10 000	0.0022	0.0022	0.0022	0.0022	0.	
		16 000	0.0033	0.0022	0.0022	0.0022 (v)	0. (ϵ_0)	No failure
150......	2	0	0.	....	....		....	
		16 000	0.0013	....	....	<0.00070[b] (v)	....	No failure
100......	2	0	0.	....	....		....	
		16 000	0.0016	....	....	<0.0010[b] (v)	....	No failure

[a] Elongation afte: failure.
[b] Value obtained by dividing total creep by elapsed time.

NOTES:
(F) Denotes failure of test specimen after time interval indicated.
(T) Indicates time at which third stage of creep begins.
(v) Minimum tangent creep rate observed during test period.
(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

companion paper (1). The alloys varied in silver content from 0 to 0.018 per cent and in copper content from 0 to 0.061 per cent as indicated in Table I. At the start of these tests the alloys were approximately 18 months old.

METHODS OF TEST AND TEST DATA

The tensile strength and the corresponding elongation in 2 in. were determined on specimens cross-milled from flattened sections of pipe to the dimensions shown in Fig. 1. In computing tensile strengths, the cross-section of the specimens was determined from measurements made to the nearest 0.0001 in. All tension tests were made on a 600-lb. Amsler pendulum type testing machine operated at a head speed of 0.250 in. per inch per minute. The tension data obtained from these tests are tabulated in Table I and the effects of silver and copper are illustrated in Fig. 2.

TABLE VII.—CREEP CHARACTERISTICS OF LONGITUDINAL SPECIMENS CUT FROM EXTRUDED LEAD PIPE.

Alloy No. 35—0.017 per cent silver, 0.061 per cent copper. Tested at 85 F., 3-in gage length.

Stress, psi.	Number of Specimens Tested	Total Time, hr.	Total Creep, in. per inch	Creep Rate, in. per inch per 10,000 hr.			Intercept, in. per inch	Characteristic Fracture
				Average for Total Time Interval	Average for Past Interval	Tangent Rate		
2000......	1	0	0.0207	....	....	....	....	
		4.1 (F)	0.591^a	141	139	....	....	Ductile
1500......	1	0	0.0040	....	....	....	....	
		25	0.1095	43.8	42.2	....	....	
		50	0.1988	39.7	35.7	....	....	
		76 (F)	0.492^a	64.8	113	....	....	Ductile
1000......	1	0	0.	....	....	....	....	
		25	0.0034	1.36	1.36	1.06	0.0005	
		50	0.0050	1.00	0.64	0.89	0.0010	
		1 040 (T)	0.0940	0.90	0.90	0.89 (v)	0.0010 (ϵ_0)	
		1 250	0.1222	0.98	1.13	1.63	....	
		1 936 (F)	0.552^a	2.85	6.27	....	....	Ductile
800......	1	0	0.	....	....	....	....	
		100	0.0020	0.20	0.20	0.16	0.0005	
		1 000	0.0123	0.12	0.11	0.11	0.0015	
		1 620 (T)	0.0195	0.12	0.11	0.11 (v)	0.0015 (ϵ_0)	
		2 000	0.0280	0.14	0.22	0.20	....	
		7 446 (F)	0.354^a	0.47	1.02	....	....	Brittle
600......	1	0	0.	....	....	....	....	
		1 000	0.0030	0.030	0.030	0.030	0.	
		2 000	0.0060	0.030	0.030	0.030	0.	
		5 000	0.0150	0.030	0.030	0.030	0.	
		10 000	0.0300	0.030	0.030	0.030	0.	
		13 500 (T)	0.0400	0.030	0.030	0.030 (v)	0. (ϵ_0)	
		16 000	0.0505	0.032	0.042	0.045	....	No failure
400......	2	0	0.	....	....	....	....	
		1 000	0.0015	0.015	0.015	0.015	0.	
		2 000	0.0028	0.014	0.013	0.012	0.0005	
		5 000	0.0067	0.013	0.013	0.0088	0.0023	
		7 500	0.0094	0.013	0.011	0.0079	0.0035	
		10 000	0.0114	0.011	0.0080	0.0079	0.0035	
		16 000	0.0162	0.010	0.0080	0.0079 (v)	0.0035 (ϵ_0)	No failure
250......	2	0	0.	....	....	....	....	
		5 000	0.0019	0.0038	0.0038	0.0038	0.	
		7 500	0.0028	0.0037	0.0036	0.0035	0.0002	
		10 000	0.0036	0.0036	0.0032	0.0016	0.0020	
		16 000	0.0046	0.0029	0.0017	0.0016 (v)	0.0020 (ϵ_0)	No failure
150......	2	0	0.	....	....	....	....	
		10 000	0.0013	0.0013	0.0013	0.0010	0.0003	
		16 000	0.0019	0.0012	0.0010	<0.0010 (v)	0.0003 (ϵ_0)	No failure
100......	2	0	0.0007	....	....	....	....	
		16 000	0.0017	0.0011	0.00062	<0.00062 (v)	0.0007 (ϵ_0)	No failure

[a] Elongation after failure.

NOTES:

(F) Denotes failure of test specimen after time interval indicated.

(T) Indicates time at which third stage of creep begins.

(v) Minimum tangent creep rate observed during test period.

(ϵ_0) Strain intercept at zero time obtained by extrapolation of tangent.

The creep tests were made on tension specimens (Fig. 1) using a 3 in. gage length and following the procedure outlined in the previous paper by Gohn, Arnold, and Bouton (6). All tests were made at a temperature of 80 to 85 F. The precision of the strain measurements was ± 0.0007 in. per inch for the 3 in. gage length used. The original elongation-time data obtained in these studies are on file at the A.S.T.M. headquarters. Derived data recorded in Tables II to

VII inclusive at specified time intervals were obtained by interpolation between the bracketing observations.

age creep rate for any given time interval. The second (column 6) represents the average creep rate occurring be-

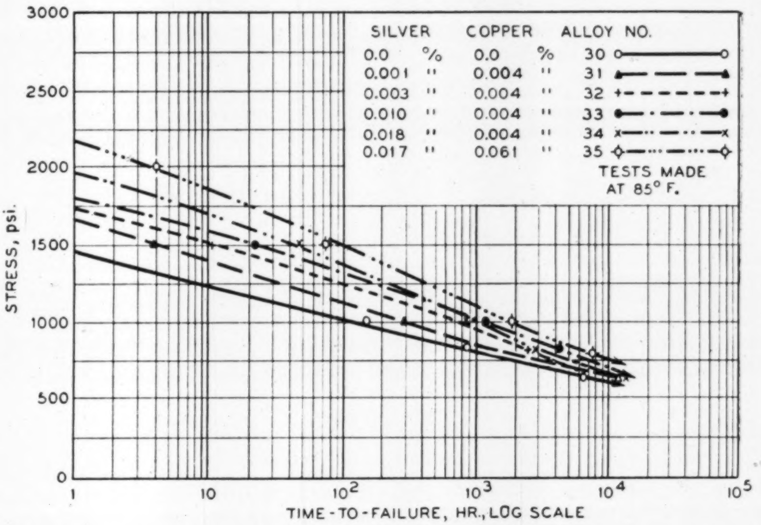


FIG. 3.—Summary of Stress Rupture Data for Lead-Silver Cable Sheathing Alloys.

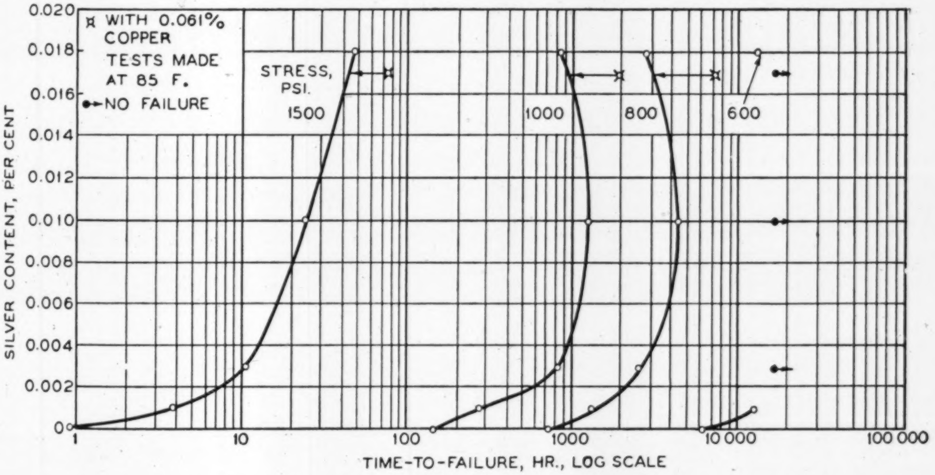


FIG. 4.—Relation Between Silver Content and Time-To-Failure (Stress Rupture).

The elongation-time data have been analysed according to the plan of presentation suggested by the A.S.M.E.-A.S.T.M. Joint Research Committee (7), and the results of this analysis are given in terms of creep rates in Tables II to VII. Three values are given. The first (column 5) represents the average creep rate for any given time interval. The second (column 6) represents the average creep rate occurring between two successive time intervals while the third (column 7) represents the tangent creep rate. The latter is obtained by drawing the best smooth, conventional creep curve through the observed time-elongation data (plotted to Cartesian coordinates). From such curves it is evident that the tangent

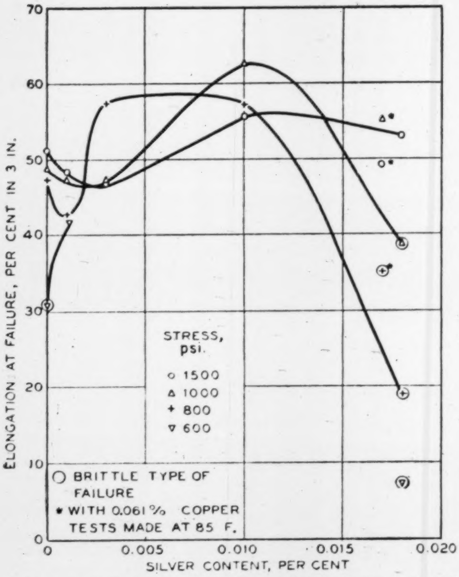
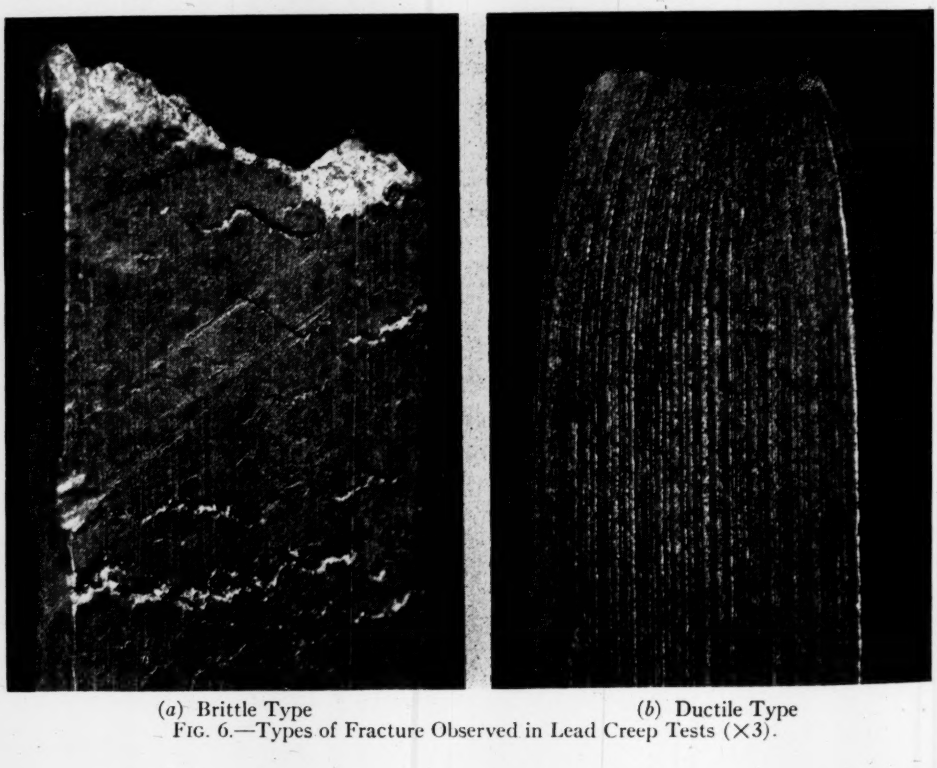


FIG. 5.—Relation Between Silver Content and Elongation at Failure (per cent in 3 in.).

creep rates decrease with time during the first stage of creep, become constant during the second stage, and increase during the third stage of creep until failure occurs. It is therefore evident that the tangent creep rates (v) are at a minimum during the second stage of creep. These minimum creep rates together with their associated zero time intercepts (ϵ_0), obtained by extrapolating the tangent back to zero time, are widely used in design applications involving creep.

DISCUSSION OF DATA

Figure 2 shows that, for a constant copper content, the tensile strength increases with increasing silver content. The data do not admit of showing the effect of copper alone in strengthening lead. However, when the silver content was constant at 0.017 to 0.018 per cent,



(a) Brittle Type (b) Ductile Type

FIG. 6.—Types of Fracture Observed in Lead Creep Tests (×3).

an increase in the copper content from 0.004 to 0.061 per cent resulted in an increase of only 120 psi. in tensile strength and a slight increase in elongation.

The stress-rupture data shown in Fig. 3 indicate that at high stresses, the time to failure increases with increasing silver content. At lower stresses, the curves tend to converge, indicating that the effect of silver is less at these stress levels.

The effect of the added silver is more evident when the data are plotted as shown in Fig. 4 which shows the relation between silver content and time to failure at various stress levels. While at 1500 psi. the improvement in life is approximately proportional to the silver content, at lower stresses such as 1000 and 800 psi., a decrease in life resulted from the addition of silver in excess of 0.010 per cent. This is interpreted to be the result of an embrittling effect of silver in higher percentages on specimens stressed at these creep loads. This is substantiated by the lower elongation shown in Fig. 5 for the 0.018 per cent silver alloy and the "brittle type" failures observed at these intermediate stresses (Tables II to VII).

It is of interest that the addition of copper to the high silver alloy nullifies this embrittling effect to a degree as shown by the comparatively longer life (Fig. 4) and improved elongation (Fig. 5) of the alloy containing 0.061 per cent of copper.

The term "brittle type of failure," as used in this discussion is applied to a fracture in which there is little or no visual evidence of local contraction of area. This is illustrated in Fig. 6 (*a*). On the other hand the term, "ductile type of failure" is applied to a fracture in which there is marked local elongation at the fracture with a considerable reduction in area. This is shown in Fig. 6 (*b*). While some lead alloys are really brittle in that they fail with little or no elongation, a brittle type failure is not necessarily associated with a low elongation (Fig. 5). The explanation for this lies in the mechanism of failure for lead under creep loads. While at high stresses a typical creep failure resembles the ductile type of failure shown in Fig. 6 (*b*), at creep stresses below some

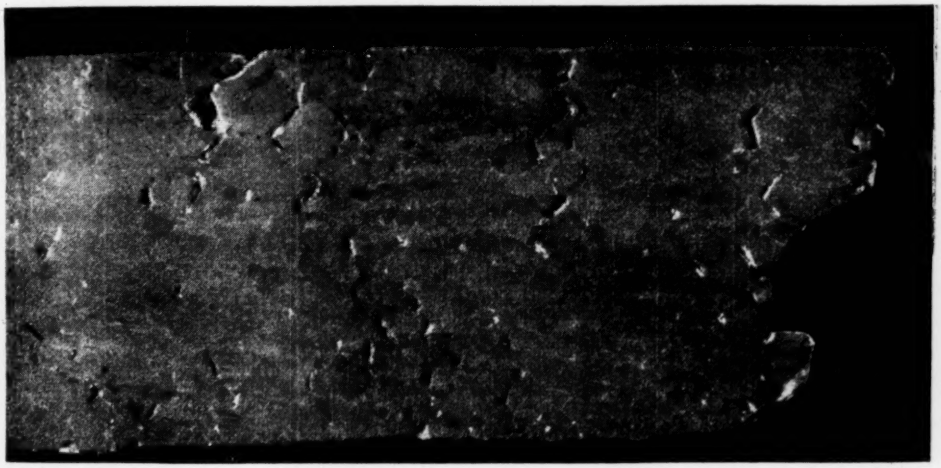


FIG. 7.—Section at Fracture of 0.018 Per Cent Silver-Lead Alloy (No. 34) Showing Grain Boundary Separation. Failed at 800 psi. in 2786 hr. with 19.1 per cent elongation in 3 in. (×4).

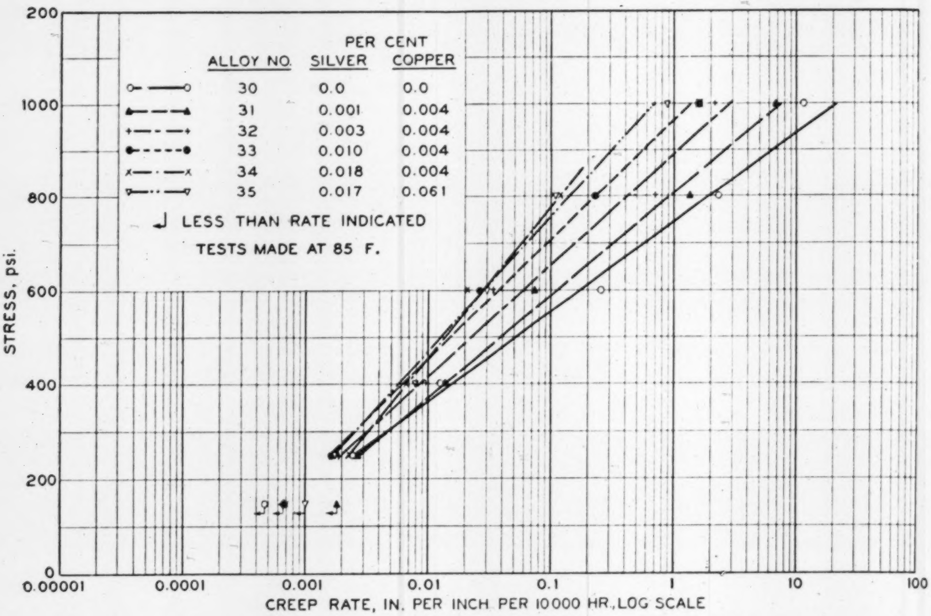


FIG. 8.—Relation Between Creep Rate and Stress for Lead-Silver Cable Sheathing Alloys Supplied by Philadelphia Electric Co.

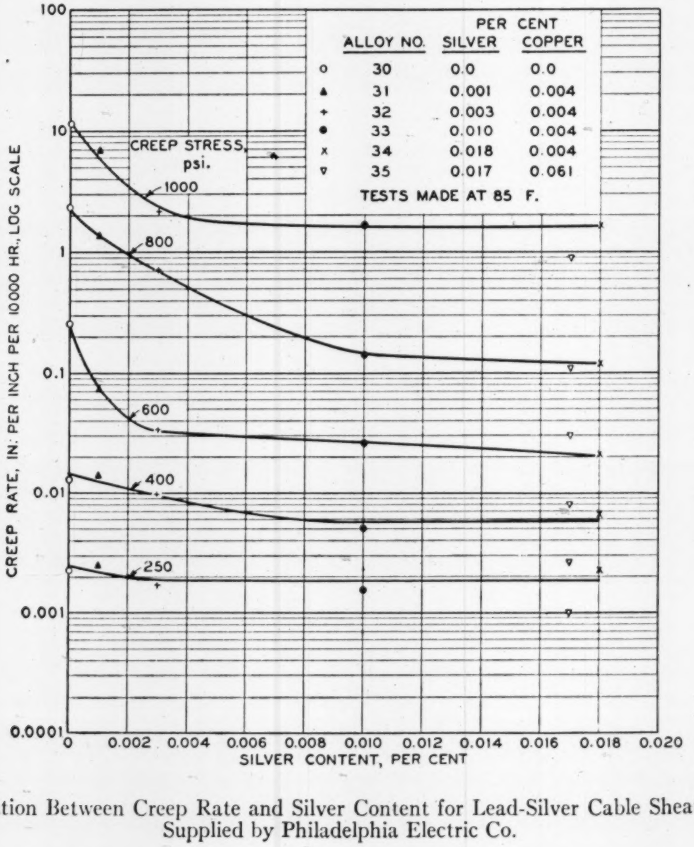


FIG. 9.—Relation Between Creep Rate and Silver Content for Lead-Silver Cable Sheathing Alloys Supplied by Philadelphia Electric Co.

critical value there appears to be a change in the mechanism of creep and the type of failure becomes predomnantly brittle as illustrated by Fig. 6 (*a*). A typical microsection of a brittle failure in silver bearing lead is shown in Fig. 7. This micrograph shows a section at the fracture of a specimen of alloy No. 34 which failed at a stress of 800 psi. after 2786 hr. with an elongation of 19.1 per cent. Localized rupture has occurred throughout the section as a result of grain boundary separation.

Voids are sometimes observed in ductile fractures of lead alloys under creep loads but appear to be absent in fractures resulting from short-time tension tests. The authors are not prepared, at this time, to offer an explanation for the differences observed in fracture under short-time tension and steady creep loads but are now obtaining more evidence on this subject.

Since the creep rate is the controlling factor in many applications of lead alloys, the effect of silver on this important property was analyzed. Figure 8 shows that at high stress levels, the creep rate is substantially decreased by increasing percentages of silver. Like the stress-rupture curves (Fig. 3), the rate curves tend to converge at lower stress levels, again indicating that the effect of silver on creep resistance is less at these stress levels.

The effect of silver is more evident when the data are plotted as shown in Fig. 9 which shows the relation between silver content and creep rate at various stress levels. As in the case of the stress-rupture curves shown in Fig. 4, the major effect on creep occurs for the first 0.010 per cent of silver. At stresses of 1000 to 600 psi. the rate of creep was reduced by a factor of approximately 10 by the addition of this amount of silver. The improvement at 400 psi. was only about one-fourth that observed at the higher stresses while at 250 psi. the effect of silver was negligible. At lower stresses the effect of silver was indeterminate after 16,000 hr. However, the convergence of the creep rate-stress curves (Fig. 8) at low stress indicates that the effect of silver at stresses below 250 psi. is small. No appreciable decrease in the creep rate was observed as a result of the addition of silver in amounts greater than 0.010 per cent. The effect of 0.061 per cent of copper on the creep rate of the alloy containing 0.017 per cent of silver was minor. These findings are generally the same as those reported by Russell (2) from tests made on cast-and-rolled specimens.

These findings on creep are important not only with respect to the application of lead alloys for cable sheathing under conditions of continuously applied load, but also in applications involving fatigue. When subjected to fatigue loading the lead sheath is stressed in the plastic range by repeated bending. Since this is a specialized type of creep it would, therefore, appear likely that there is some relation between the bending fatigue characteristics of lead alloys stressed in the plastic range and their creep properties. The authors are therefore investigating the fatigue properties of these alloys in bending, but such tests are at present incomplete.

CONCLUSIONS

Silver up to 0.010 per cent markedly decreased the creep rate of lead at stresses of 400 psi. and above. The effect was less at 400 psi. than at higher stresses. At stresses of 250 psi. and below the data indicate that the effect of silver on the creep rate is small. The addition of more than 0.010 per cent of silver or the addition of 0.061 per cent of copper to silver bearing lead containing 0.017 per cent of silver had only a small effect on the creep rate at all stresses. The data show that for the complete evaluation of the creep characteristics of alloys, stress-rupture curves should be accompanied by creep rate data. The data further show the importance of the choice of stress used for conducting creep tests and its effect upon creep rates. Wherever possible, creep rates should be obtained for the stresses of interest from test data rather than by extrapolation from data taken at higher stress levels.

Acknowledgments:

The authors are appreciative of the valuable discussions of the interpretation of these data with Messrs. H. S. Phelps, F. Kahn, and W. P. Magee. Messrs. F. G. Foster and R. M. Sample of the Research Department of the Bell Telephone Laboratories, Inc., prepared the micrographs used in this paper.

REFERENCES

(1) H. S. Phelps, F. Kahn, and W. P. Magee, "Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing," see p. 815 of this publication.

(2) R. S. Russell, "Influence of Impurities on the Properties of Lead. Part V. Creep Tests on Electrolytic Lead and Some of Its Alloys," *Proceedings*, Australasian Inst. of Mining and Metallurgy, (NS) No. 101, p. 33 (1936).

(3) J. N. Greenwood and H. K. Worner, "Type of Creep Curve Obtained with Lead and Its Alloys," *Journal*, Inst. of Metals, Vol. 64, p. 135 (1939).

(4) J. N. Greenwood and J. H. Cole, "Creep Rate of Various Industrial Leads," *Metallurgia*, Vol. 36, p. 233 (1947).

(5) A. J. Phillips, "Some Creep Tests on Lead and Lead Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 170 (1936).

(6) G. R. Gohn, S. M. Arnold, and G. M. Bouton, "Creep Tests on Some Extruded Lead and Lead-Alloy Sleeves and Tapes," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 990 (1946).

(7) Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys, Am. Soc. Testing Mats., and Am. Soc. Mechanical Engs. (1938). (Issued as separate publication, *STP No. 37*).

[**See Joint Discussion, p. 825.**—ED.]

INFLUENCE OF SMALL PERCENTAGES OF SILVER ON THE TENSILE STRENGTH OF EXTRUDED LEAD SHEATHING*

By Howard S. Phelps,[1] Frank Kahn,[1] and William P. Magee[1]

Synopsis

Stress-rupture tests were made on a series of extruded cable sheathing pipe samples of substantially pure lead to which various percentages of silver were added as metallic additions (up to 0.018 per cent silver). Extrapolation of results indicates probability of improved stress-rupture and creep characteristics at operating stresses when silver up to approximately 0.010 per cent is added to pure lead. Undesirable results are indicated for addition of silver alone in excess of 0.010 per cent.

Lead used for sheaths of electric power cables is required by the specifications of the Association of Edison Illuminating Companies (**1**)[2] to be commercially pure lead conforming to certain grades of lead of not less than 99.85 per cent purity specified in A.S.T.M. Standard Specifications for Pig Lead (B 29 – 43).[3] The component metallic elements other than lead present in these A.S.T.M. grades vary with the source of the ores and the method of refining. Although these metallic additions are present in very small percentages, it is well known that they have a disproportionately large influence on the physical characteristics of sheathing extruded from these leads.

For a number of years Philadelphia Electric Co. has been carrying on long-time constant-pressure bursting tests of full-section specimens of cable sheathing of various manufacture (**2**). For the same initial stress in the various samples tested the time-to-burst was found to vary over a considerable range. When an attempt was made to correlate these data with the percentages of the elements commonly present as minor components, it became evident that the results were predominantly influenced by the percentage of silver present.

These results were discussed with several cable manufacturers who were invited to cooperate in the study by supplying extruded cable sheathing pipes of lead containing various percentages of silver. Through the kindness of The Okonite-Callender Cable Co., Inc., pipe samples of six compositions were supplied for bursting tests. Also, through the kindness of the General Electric Co., cast-and-rolled strips of four compositions were supplied for comparative strip-tension tests.

Portions of the sheathing pipe samples were made available to Bell Telephone Laboratories, Inc., for a parallel investigation of creep and bending fatigue characteristics. The results of the creep tests by that organization are being presented in a companion paper at this meeting, entitled, "The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead," by G. R. Gohn and W. C. Ellis (3).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Engineer of Division, Engineer of Special Tests Branch, and Special Tester, respectively, Special Investigation and Testing Division, Philadelphia Electric Co., Philadelphia, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 824.

[3] 1946 Book of A.S.T.M. Standards, Part I-B, p. 199.

MATERIALS INVESTIGATED

The samples of extruded lead cable sheathing pipe supplied by The Okonite-Callender Cable Co., Inc., and studied in the present investigation consisted of six compositions. Sample No. 30 was extruded from high purity lead. Samples Nos. 31, 32, 33 and 34 were extruded from melts prepared by adding a 2.5 per cent silver-lead master alloy to the same high purity lead, and sample No. 35 by adding the 2.5 per cent silver-lead alloy and a 5 per cent copper-lead alloy to the high purity lead.

TABLE I.—CHEMICAL COMPOSITION OF LEAD SHEATHING PIPE SAMPLES.

Phila. Electric Co. Sample	Composition, per cent[a]				
	Silver	Copper	Bismuth	Sodium[b]	Lead[c]
No. 30......	0	0	0.004	0.001	99.995
No. 31......	0.001	0.004	0.005	0.002	99.988
No. 32......	0.003	0.004	0.005	0.002	99.986
No. 33......	0.010	0.004	0.005	0.002	99.979
No. 34......	0.018	0.004	0.005	0.002	99.971
No. 35......	0.017	0.061	0.005	0.002	99.915

[a] Chemical composition according to The Okonite-Callender Cable Co., Inc.

[b] Sodium added by the manufacturer in treating the lead.

[c] Lead by difference.

Table I shows the chemical composition of the six samples of sheathing pipe.

In fabricating the various pipes, the melts were treated with a sodium-lead alloy for chemical stabilization. The pipe was extruded at a rate of approximately 30 ft. per min. from a Robertson-type press using the Hill Standpipe method of filling the lead cylinder from the bottom. Immediately after extrusion, the pipe was chilled with a fine spray of water.

These pipes were approximately all of the same sectional size, averaging 2.65 in. in outside diameter and 0.143 in. in thickness.

The cast-and-rolled lead strips supplied by General Electric Co. and included in this study consisted of four compositions. Sample No. 1 was cast from exceptionally pure lead. Samples Nos. 2, 3, and 4 were cast from melts prepared by adding to this exceptionally pure lead a silver-lead master alloy made from the same lead.

Table II gives the composition of the lead strip samples, based on a chemical analysis typical of the grade of exceptionally pure lead from which the samples were prepared.

TABLE II.—CHEMICAL COMPOSITION OF CAST-AND-ROLLED STRIP SAMPLES.

Phila. Electric Co. Sample	Composition, per cent[b]					
	Silver	Copper	Cadmium	Iron	Zinc	Lead[c]
No. 1[a] ...	0.0002	0.0002	0.0005	0.0002	0.0001	99.9988
No. 2.....	0.0022	0.0002	0.0005	0.0002	0.0001	99.9968
No. 3.....	0.0052	0.0002	0.0005	0.0002	0.0001	99.9938
No. 4.....	0.0102	0.0002	0.0005	0.0002	0.0001	99.9898

[a] American Smelting and Refining Co. Alton brand lead.

[b] Chemical composition according to the General Electric Co. based on a chemical analysis supplied by the producer and stated to be typical of Alton brand lead. Bismuth, antimony, nickel, cobalt, arsenic, and tin, nil.

[c] Lead by difference.

In fabricating the strip samples, bars 1 by 1.5 by 9 in. were cast from the several melts and machined to obtain clean surfaces. The bars were then rolled to a thickness of $\frac{1}{8}$ in. The strip samples, as supplied, were approximately 18 in. long, 2 in. wide and $\frac{1}{8}$ in. thick.

METHODS OF TEST

The constant internal pressure hydraulic bursting tests were made at a substantially constant temperature of 110 F. on cable pipe specimens approximately 12 in. long with plugged ends, and with pressure supplied from an accumulator having an air cushion over a column of water, in the manner described by Phelps, Gates, and Kahn (2).

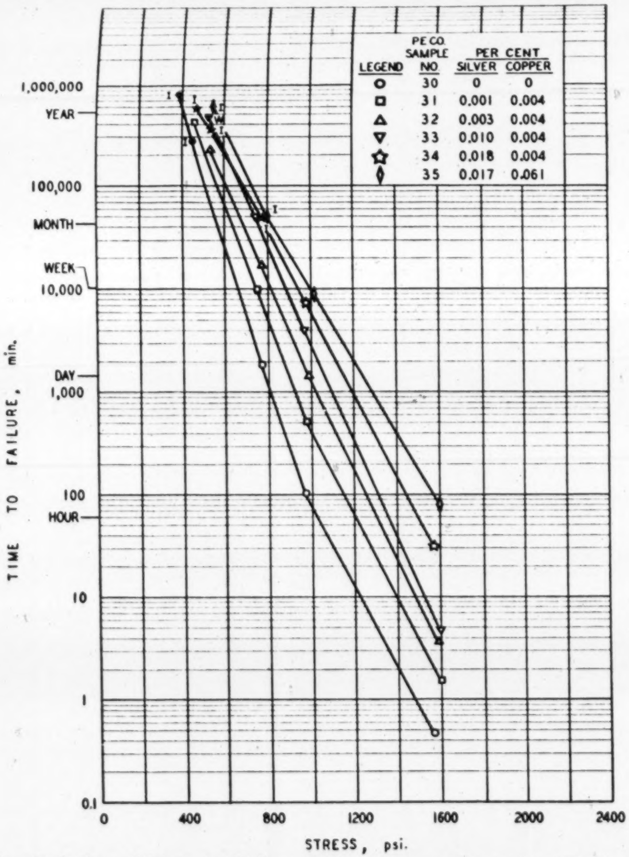


FIG. 1.—Relation of Time-to-Failure Versus Stress for Bursting Tests of Lead Sheathing Pipe at 110 F.

Open characters indicate necked-down failures. Solid characters indicate radia failures. The letter *I* denotes intercrystalline failure and *W* weld failure.

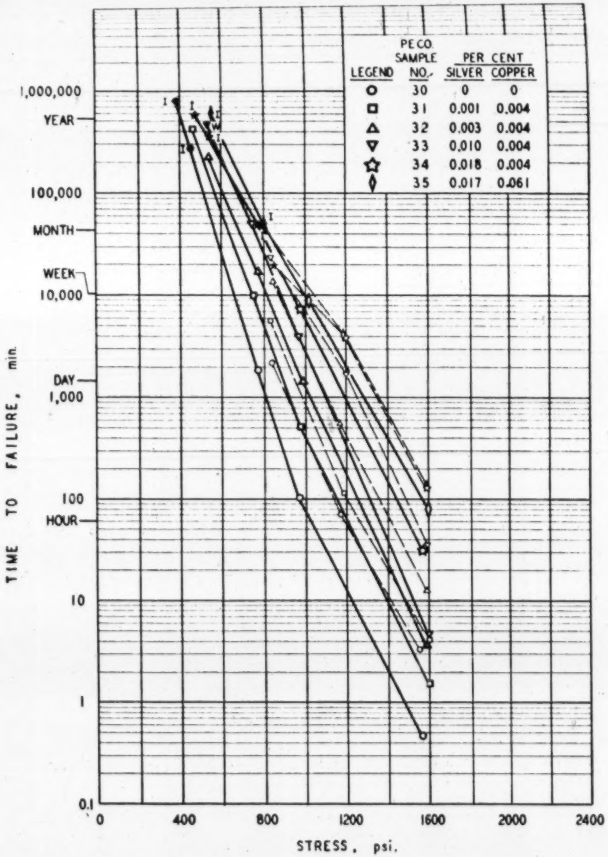


FIG. 2.—Comparison of Stress-Rupture Curves at 110 F.

Solid lines are for bursting tests. Dashed lines are for strip-tension tests. Open characters indicate necked-down failures. For the cylindrical specimens, solid characters indicate radial failures. The letter *I* denotes intercrystalline failure and *W* weld failure. For the strip-tension specimens, solid characters indicate intercrystalline failures.

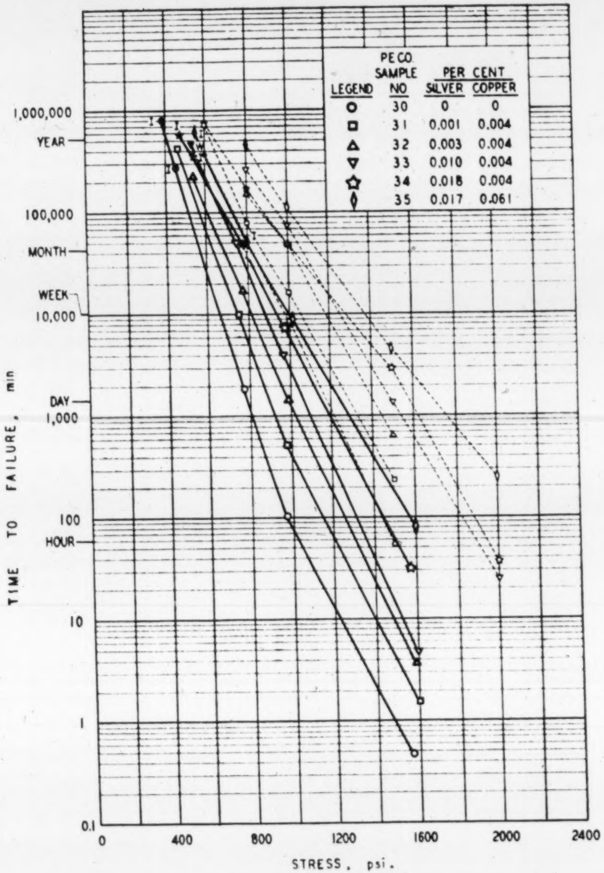


FIG. 3.—Comparison of Stress-Rupture Curves.

Solid Lines are for bursting tests at 110 F. of Fig. 1. Dotted lines are for strip-tension tests at 80–85 F. by Gohn and Ellis. Open characters indicate necked-down failures. For the cylindrical specimens, solid characters indicate radial failures. The letter *I* denotes intercrystalline failure and *W* weld failure. For the strip-tension specimens, solid characters indicate intercrystalline failures.

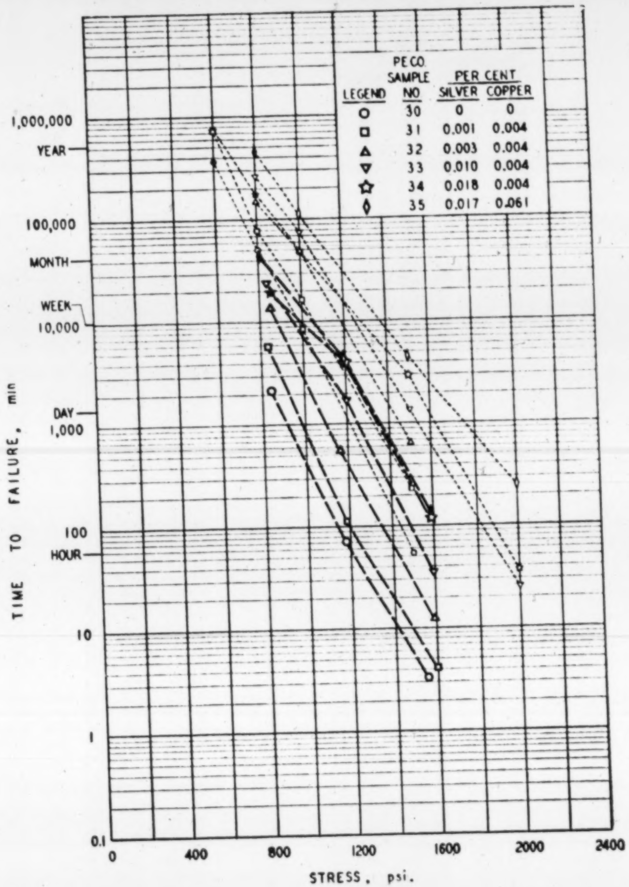


FIG. 4.—Comparison of Stress-Rupture Curves.

Dashed lines are for strip-tension tests at 110 F. Dotted lines are for strip-tension tests at 80–85 F. by Gohn and Ellis. Open characters indicate necked-down failures. Solid characters indicate intercrystalline failures.

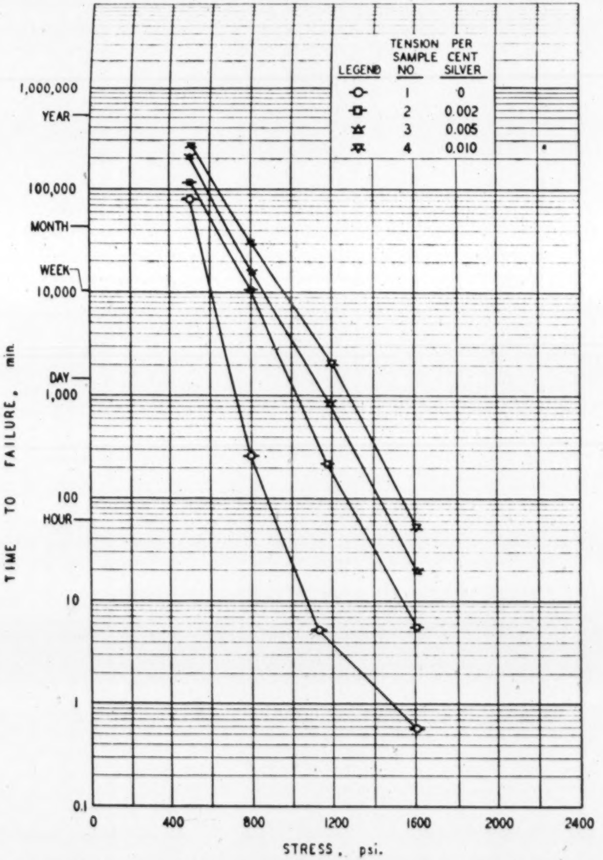


FIG. 5.—Relation of Time-to-Failure Versus Stress for Strip-Tension Tests at 110 F. of Specially Prepared Cast-and-Rolled Lead Strip Samples.

Open characters indicate necked-down failures. Solid characters indicate intercrystalline failures.

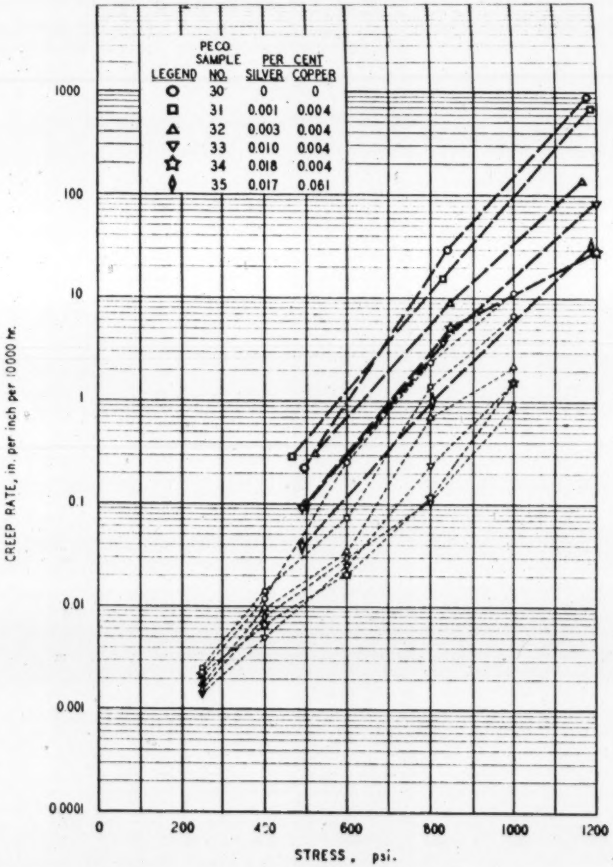


FIG. 6—Comparison of Creep Rate Versus Stress Curves.

Dashed lines are for strip-tension tests at 110 F. Dotted lines are for strip-tension tests at 80–85 F. by Gohn and Ellis.

The constant tension tests were made on specimens stamped from both the strip samples and from flattened longitudinal sections of the pipe samples with a steel die designed for punching out 2-in. gage length rubber tension-test specimens $\frac{1}{4}$ in. wide in accordance with A.S.T.M. methods, Standard Methods of Tension Testing of Vulcanized Rubber (D 412 - 41).[4] When pressed at low speed in a testing machine through the lead strips backed with hard wood, this die produced a specimen of excellent appearance with very slight deformation of the crystal structure along the sheared edges. The cast-and-rolled strip specimens were annealed for one hour at 250 C. before test.

The tension specimens, loaded with dead weights in a manner similar to the tests of Moore and Dollins (4), were enclosed in a box maintained at a temperature of 110 F. ± 1 F. and viewed through a glass window.

The 2-in. gage length of each specimen was marked off at $\frac{1}{2}$-in. intervals by a scratched mark in a narrow transverse stripe of varnish. A scale graduated to $\frac{1}{64}$ in. was attached to the top of the specimen and disposed in close proximity to register with the scratched marks. Readings of creep were taken through a magnifying glass to give an accuracy of approximately 0.004 in.

RESULTS OF TESTS

Figure 1 shows curves of time-to-failure at 110 F. *versus* stress (stress-rupture curves) for the bursting tests of the sheathing pipe. The stress referred to in these curves is the initial true maximum circumferential stress obtained by calculation from the dimensions of the test specimen according to a formula developed at the Philadelphia Electric Co. and discussed by Phelps, Gates, and Kahn (2). This formula for maximum circumferential stress is stated as follows:

$$S = PD/2t_{min.} + P\ (1.0625 - 0.1125\,D/t_{avg.})$$

where:

P = internal gage pressure (held constant),

D = initial internal diameter,

$t_{min.}$ = initial minimum thickness, and

$t_{avg.}$ = initial average thickness.

It is to be observed that the foregoing formula is substantially the conventional hoop-stress formula for a thin-walled cylinder, $S = \dfrac{PD}{2t}$, with a correction factor for the effect of wall thickness.

Figure 2 compares the bursting test stress-rupture curves of Fig. 1 with similar curves for strip-tension tests of the same sheathing samples, all at a temperature of 110 F.

Figure 3 compares the bursting test stress-rupture curves at 110 F., shown in Fig. 1, with stress-rupture curves for strip-tension tests at 80 to 85 F. on specimens cut from the same sheathing samples, from test data presented by Gohn and Ellis (3).

Figure 4 compares the strip-tension stress-rupture curves at 110 F. by the Philadelphia Electric Co. with the similar curves for the same samples at 80 to 85 F. by Gohn and Ellis (3).

Figure 5 shows strip-tension stress-rupture curves for the four cast-and-rolled samples tested at 110 F.

Figure 6 compares curves of creep rate *versus* stress for the sheathing pipe strip-tension specimens, tested at 110 F. by the Philadelphia Electric Co., and at 80 to 85 F. by Gohn and Ellis (3).

DISCUSSION OF RESULTS

Stress Rupture Data:

The stress-rupture curves of Fig. 1, all at a controlled temperature of 110 F., indicate that the increase in life of the samples containing silver over that for the silver-free sample was roughly proportional to the silver content. At stresses above approximately 1000 psi., the ratio of increase in life was roughly equal to 4000 times the percentage of silver. For example, above 1000 psi., sample No. 34 (0.018 per cent silver) had a life greater than that of the silver-free sample No. 30 by a factor of approximately $4000 \times 0.018 = 72$.

---

[4] 1946 Book of A.S.T.M. Standards, Part III-B, p. 247.

The failures at stresses above 1000 psi. were of the necked-down type, although the necking was only partial with evidence of intercrystalline cracks for the specimens from samples Nos. 34 and 35 at approximately 1000 psi.

At stresses of the order of 500 psi., there was still an apparent gain in life attributable to the silver content, but this gain was reduced to the order of only $\frac{1}{10}$ of that observed at stresses above 1000 psi. In the tests at approximately 500 psi. stress, the bursts for samples Nos. 31 and 32, containing 0.001 per cent and 0.003 per cent silver, respectively, were still of the necked-down type, whereas the bursts for samples with greater percentages of silver were of intercrystalline type.

In order to determine whether bursts which occurred without necking down should be classified as intercrystalline or transcrystalline type failures, transverse sections adjacent to the bursts were polished and etched with ammonium molybdate etchant. If definite intercrystalline cracks appeared, the failure was classified as of intercrystalline type.

Results for extruded sheathing at stresses of the order of 500 psi. show that silver, when present in the proportion of at least 0.010 per cent, apparently reduces the grain boundary strength so that failure is by intercrystalline rather than necked-down fracture. The data indicate that the equicohesive stress at 110 F. is relatively higher for a silver content of 0.010 per cent or more than for a silver content of 0.003 per cent or less.

An interesting development was the abrupt change in slope of the stress-rupture characteristic of sample No. 34 (0.018 per cent silver) at a stress of approximately 800 psi. For stresses above 800 psi., the slope of the stress-rupture characteristic appeared consistent with those for the other samples, whereas for stresses below this value, a decreased negative slope was indicated. This decreased negative slope at the lower stresses is a distinctly undesirable trend which, if continued to the low values of stress at which cable sheathing is normally operated, indicates sheathing of this composition would have a relatively short life. It is to be observed that the curve for sample No. 34 has crossed the curve for sample No. 33 (0.010 per cent silver). Although sample No. 35 (0.017 per cent silver, 0.061 per cent copper) contains substantially the same proportion of silver as sample No. 34, the curve for sample No. 35 does not exhibit this undesirable change in slope. If this proportion of silver is above the solid solubility limit in lead, the foregoing difference in behavior might be explained by the possibility that the silver and copper, which form binary solid solutions, are in ternary solution with the lead in sample No. 35.

Figure 2, comparing the results of the bursting tests and the strip-tension tests of the same sheathing samples, all at a temperature of 110 F., shows that the strip-tension tests corroborate the life-increasing influence of the silver content, which was indicated by the bursting tests. However, the curves for the strip specimens, as a group, are shifted upwardly on the scale of ordinates as compared with the pipe specimens, indicating the strip specimens have a generally greater life for equal stress. It seems reasonable to assume that the difference in time-to-failure for the two types of test is due to the fact that the cylindrical specimens were subjected to triaxial stresses (longitudinal tension approximately 50 per cent of circumferential stress and radial compression approximately 10 per cent of circumferential stress) whereas the strip specimens were in simple tension. The strip-tension data show an even more pronounced decrease in negative slope for sample No. 34, the change apparently occurring at approximately 1200 psi.

In Fig. 3, the stress-rupture curves of Fig. 1 for bursting tests at 110 F. are compared with the stress-rupture data of Gohn and Ellis (3) for the strip-tension tests at 80 to 85 F. on specimens cut from the same sheathing pipe samples. The strip-tension tests at 80 to 85 F. and the bursting tests at 110 F. show a very similar discrimination or separation of the curves on the basis of silver content of the samples. On the other hand, the strip-tension curves, as a group, are positioned approximately one cycle higher on the scale of ordinates, indicating an increase in life of ten-fold order. This difference in results is apparently attributable to the difference in temperature at which the two series of tests were conducted and also to the difference in distribution of stresses. The strip-tension data at 80 to 85 F. again show the change in slope for sample No. 34, which appears to occur at 1500 psi.

In Fig. 4 the strip-tension tests at 110 F. at Philadelphia Electric Co. are compared with those by Gohn and Ellis (3) at 80 to 85 F. to show the increase in life attributable to reducing the temperature from 110 F. to 80 to 85 F. The curves for the tests at the lower temperature show that the increase in life obtained by lowering the temperature is still of the order of ten fold. Aside from the difference in temperature, the test procedures differed only in shapes of the test specimens and their methods of preparation. (The Philadelphia Electric Co. strip-tension test specimens were $\frac{1}{4}$ in. wide and of 2 in. gage length pressed out with a die, whereas those of Gohn and Ellis (3) were machined specimens $\frac{1}{2}$-in. wide and of 3-in. gage length.) The influence of these differences in the specimens is considered negligible and the results are believed to indicate substantially the effect of the difference in test temperatures.

The stress-rupture curves at 110 F. for the cast-and-rolled strip-tension specimens shown in Fig. 5 illustrate a discrimination on the basis of silver content very similar to that of the extruded sheathing specimens. It is of interest to note that necked-down failures occurred for sample No. 1 (pure lead without silver) for stresses as low as 500 psi., whereas intercrystalline failures occurred at 800 psi. for samples Nos. 2, 3, and 4 comprising the same lead containing 0.002, 0.005 and 0.010 per cent silver, respectively. The equicohesive stress at 110 F. appears to be appreciably higher for specimens from cast-and-rolled samples than from extruded samples of similar silver content.

Creep Data:

Figure 6 shows curves of creep rate *versus* stress for the tension tests of strip specimens cut from the extruded sheathing pipe samples. The dashed curves represent results of tests at 110 F. by the authors and the dotted curves are derived from the test data of Gohn and Ellis (3) at 80 to 85 F. The creep rate in each case is the slope of the intermediate stage of constant creep of the strain-time curve plotted to Cartesian coordinates.

In the 110 F. tests at approximately 1200 psi., there was a consistent decrease in the creep rate with increasing silver content, as indicated by the vertical separation of the curves at that stress. As the stress was decreased from approximately 1200 to 500 psi., the expected decrease in creep rate with decreasing stress was evidenced, with all the curves except those for sample No. 30 (silver-free) and sample No. 34 (0.018 per cent silver) having substantially the same general slope and maintaining the same relative separation as at 1200 psi.

The curve for the silver-free sample No. 30 follows the general slope of the other curves between 1200 and 800 psi., then increases in slope to cross the curve for sample No. 31 (0.001 per cent silver) and intersect the curve for sample No. 32 (0.003 per cent silver).

The curve for sample No. 34 (0.018 per cent silver) has considerably less slope than the general group between 1200 and 800 psi., and is crossed by the curves for sample No. 35 (0.017 per cent silver and 0.061 per cent copper) and for sample No. 33 (0.010 per cent silver). Between 800 and 500 psi., the curve for sample No. 34 closely parallels that for sample No. 33.

At 500 psi. sample No. 35 has a considerably lower creep rate than sample No. 34, although they have substantially the same proportion of silver. This difference in behavior appears attributable to the presence of the 0.061 per cent of copper in sample No. 35, paralleling the improvement of sample No. 35 over sample No. 34 under the stress-rupture tests previously discussed. It appears desirable to investigate the possibility that further improvement of the creep characteristic might be obtainable by increasing the silver content of sample No. 35.

The creep rate data of Gohn and Ellis (3) at 80 to 85 F., illustrated by the dotted lines of Fig. 6, show a quite similar trend of the influence of silver content.

A point of major interest is the influence of temperature on the creep rates. Comparison of creep rates at 110 F. (dashed curves) with those at 80 to 85 F. (dotted curves) indicates convergence of the two sets of curves with decreasing stress. At 1000 psi. the ratio of the creep rates at 110 F. to those at 80 to 85 F. is of the order of 10 to 1, whereas at 500 psi. this ratio has decreased to the order of 5 to 1.

Grain Size of Extruded Sheathing Pipe Samples:

Grain size determinations were made on the six sheathing pipe samples shortly after they were received from The Okonite-Callender Cable Co., Inc. Ring sections removed from the ends and middle of each sample were polished and etched with ammonium molybdate etchant. The average grain sizes were determined by visual comparison of magnified images with the standard micrographs for non-ferrous alloys shown in Plate I, A.S.T.M. Tentative Methods of Preparation of Micrographs of Metals and Alloys (E 2 - 44 T).[5] Table III, shows the average grain sizes of the several pipe samples.

TABLE III.—GRAIN SIZE OF EXTRUDED SHEATHING PIPE SAMPLES.

Phila. Electric Co. Sample	Average Grain Size, mm.	Percentage of Principal Minor Constituent	
		Silver	Copper
No. 30	0.50	0	0
No. 31	0.35	0.001	0.004
No. 32	0.40	0.003	0.004
No. 33	0.35	0.010	0.004
No. 34	0.30	0.018	0.004
No. 35	0.17	0.017	0.061

Re-examination of the etched rings after storage at room temperature (70 to 90 F.) for three years indicated the average grain sizes of these samples remained unchanged over that period.

Random check tests of cylindrical specimens for time-to-burst at high stress gave results substantially duplicating those for the initial tests which were made approximately three years earlier. This was considered as indicating absence of aging effects in the extruded pipe samples.

---

[5] 1946 Book of A.S.T.M. Standards, Part I-B, p. 803.

CONCLUSIONS

1. The addition to substantially pure lead of silver up to 0.010 per cent markedly increases the life and reduces the creep rate under stress-rupture tests at stresses of 500 psi. and above.

2. Lead to which 0.018 per cent silver was added showed an undesirable trend of the stress-rupture and creep-rate *versus* stress curves. By extrapolation from the stress-rupture curves it was indicated that power cable sheathing of this composition would have a relatively short life at operating stresses. From the standpoint of life and creep rate under stress-rupture test, this percentage of silver appears to be considerably in excess of optimum.

3. The stress-rupture and creep rate characteristics of lead containing 0.017 per cent silver and 0.061 per cent copper showed improvement over those of lead containing 0.010 per cent silver only.

4. Cast lead, rolled into strips and annealed, and extruded lead sheathing pipe of comparable silver-bearing composition, appear to have quite similar stress-rupture characteristics at stresses of 500 psi. and above.

5. Comparing results of tension tests at 110 F. and at 80 to 85 F. on strip specimens cut from lead sheathing pipe, time-to-failure is substantially less and creep rate substantially greater at the higher temperature. These effects of temperature difference apparently diminish as the stress level is lowered.

Acknowledgments:

The authors acknowledge the contribution of R. J. Wiseman of The Okonite-Callender Cable Co., Inc., in supplying the extruded lead sheathing pipe samples tested. Acknowledgment is also made to Lyall Zickrick of General Electric Co., for furnishing the specially prepared lead strip samples tested. Thanks are particularly due to J. R. Townsend of Bell Telephone Laboratories, Inc., for his cooperation in authorizing a paralleling investigation of the creep characteristics of the silver-bearing lead sheathing pipes and making the test results available to the authors.

REFERENCES

(1) Specifications for Impregnated Paper-Insulated, Lead-Covered Cable, "Solid" Type (Seventh Edition), "Oil-Filled" Type (Fourth Edition), prepared by the Association of Edison Illuminating Companies, pp. 11 and 43, Reprinted May, 1947.

(2) H. S. Phelps, A. M. Gates, F. Kahn, "Internal Hydraulic Bursting Tests of Lead Cable Sheathing," *Proceedings,* Am. Soc. Testing Mats., Vol. 40, p. 885 (1940).

(3) G. R. Gohn and W. C. Ellis, "The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead," see p. 801 of this publication.

(4) H. F. Moore and C. W. Dollins, "Fracture and Ductility of Lead and Lead Alloys for Cable Sheathing," *Univ. of Illinois Engineering Experiment Station Bulletin No. 347* October 19, 1943.

JOINT DISCUSSION ON EFFECT OF SILVER IN LEAD

MR. C. W. DOLLINS[1] (*presented in written form*).—The authors of these two papers are to be congratulated for the careful research done so far on the influence of small amounts of silver and copper on the creep properties of lead. However, the many users of underground lead sheathed power cables are highly interested in the creep resistance of sheathing alloys at the lower stresses namely, 100 to 300 psi. Such data were only lightly reported in these papers. In the past twenty years of creep testing at the University of Illinois, we have found many alloys that had high tensile strength in the short-time rupture tests were no stronger and sometimes weaker when stressed near the design load than some alloys of moderate strength in the short-time tests. Also, the tensile strength, as given in the short-time high-stress tests gave in many cases an erroneous impression of the creep resistance of these alloys at lower stresses. On the other hand, many alloys with lower creep rates may have their ductility so reduced by age and strain-hardening that failure occurs in an unexpectedly short time.

We have tested an alloy, henceforth known as C-410, containing 0.01 per cent silver, 0.054 per cent copper, and 0.027 per cent bismuth at 110 F. with stresses of 100, 150, 200, 250 psi. For these test conditions, this alloy had slightly less creep resistance than two chemical leads and slightly more creep resistance than two copper leads of about equal copper content and much less silver. This fact leads me to believe that 0.01 per cent silver has little effect upon the creep resistance of copper or chemical lead near 200 psi. The embrittling effect of 0.018 per cent silver for the 1000 psi. and lower tests of Bell Laboratories indicates that little more than 0.01 per cent silver is desirable. The maximum elongations in 3 in. reported for the 0.01 per cent silver alloy for stresses of 800 psi. and above are very good. However, the elongation in tests with stresses around 400 to 600 psi. are needed before this alloy can be truly rated as cable sheathing, since many of the strain-hardening alloys have very low ductility (elongation) in the low-stress tests. And due to the more complicated stress conditions in the sheath form, the ductility is much less in the sheath tests than in the strip tests. A report of the ductility of the sheath samples in the Philadelphia Electric Co. tests would be a very desirable addition to their paper.

Our C-410 alloy had 50 per cent elongation in 1 in., 31 per cent in 2 in., at 1800 psi. and 26.5 per cent in 1 in. and 16.75 in 2 in. at 600 psi., room temperature tests. This shows considerable reduction in ductility as time for fracture is increased. The time to fracture was 1½ hr. for 1800 psi. and 13,560 hr. for 600 psi. As far as strength is concerned, our tests indicate that 0.01 per cent silver does not increase the strength of

---

[1] In Charge of Investigation, "Lead Sheath Materials," University of Illinois, Urbana, Ill.

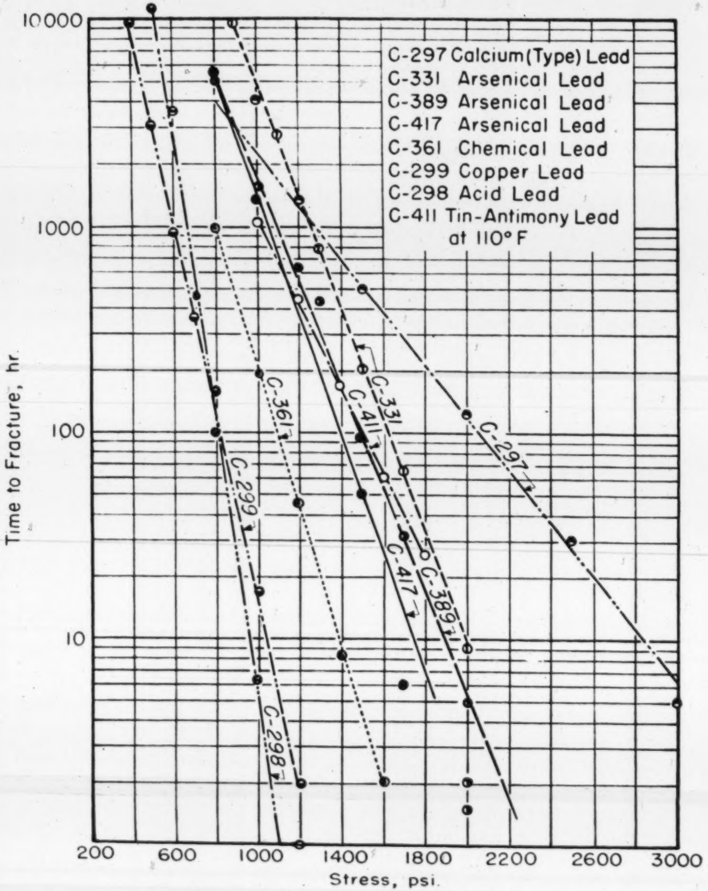


FIG. 1.—Strength-Time Diagram.

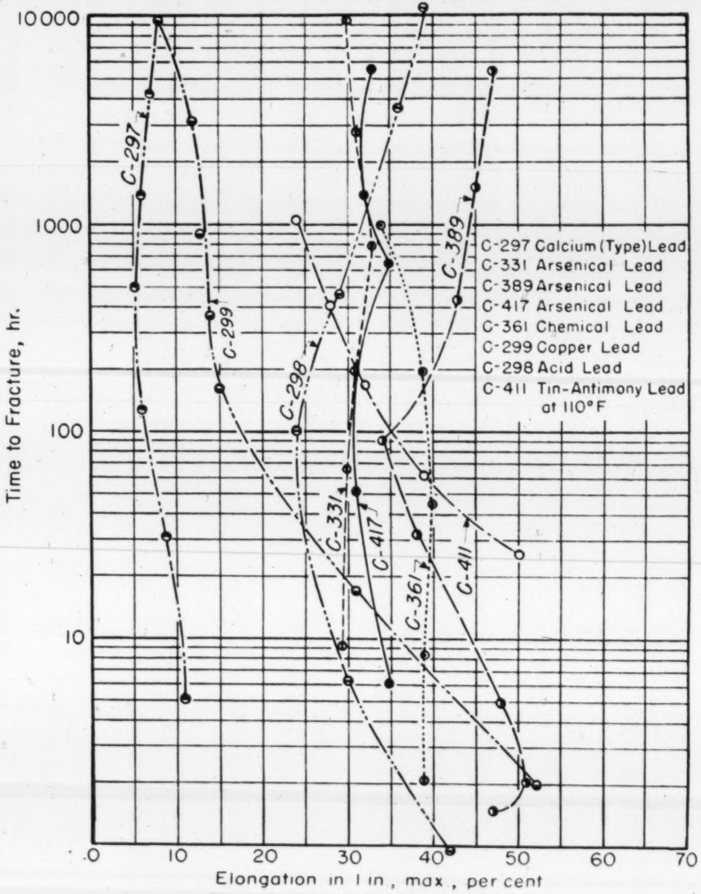


FIG. 2.—Ductility-Time Diagram.

lead very much since some chemical leads were stronger and some weaker and also some were more ductile and some less than the C-410 alloy.

The mention in the Bell Laboratories paper of the need of information on the resistance to bending of these alloys is of interest. We find that the 0.01 per cent silver alloy, C-410, has better bending resistance than some chemical and copper leads tested but has less bending resistance than most arsenical leads now being investigated for the cable industry. These arsenical leads have better creep resistance than this silver alloy at 110 F. and retain their ductility better under low-stress conditions at room temperature (about 78 F.).

(*Added Orally*).—An example of the case where a slight increase of an element in an alloy of lead greatly changes the alloy's physical properties is shown in the accompanying Fig. 1. This example of two alloys of about the same chemical content but where the bismuth was slightly increased (0.017 to 0.074 per cent) with disappointing results is shown in the data on C-298 and C-299. Alloy C-299 has considerably more strength in the short-time tests than the low-bismuth alloy C-298, but it was not long before the superior long-time strength of the low-bismuth alloy became apparent. We have tests running out past those shown in Fig. 1. The C-299 alloy, stress of 400 psi, at 110 F., withstood this load for 9700 hr. But the ductility obtained as measured by the maximum elongation in 2 in. was only 6½ per cent for that alloy, whereas the other alloy, C-298, for the same stress lasted 27,000 hr., or about three times as long, and had 23 per cent elongation in 2 in.

The cable industries are interested in the ductility of these materials which is fully as important as their strength. Only long-time tests give reliable data for either of these properties. Also shown in Fig. 1 is the chemical lead, C-361, which alloy may be considered as a representative chemical lead, in comparison with the copper lead C-299. Also shown are some arsenical alloys which have fair strength down in the range of stresses in which the cable industry is interested. The 0.02 per cent calcium alloy, C-297, containing 0.0192 per cent magnesium loses its strength quite rapidly in the tests after 1000 hr. It is believed now that the magnesium caused most of the trouble here. However, this alloy had a high strength in the short-time tests and very good creep resistance.

The ductility of these alloys is shown in the accompanying Fig. 2. The calcium-magnesium alloy, however, was very low in ductility, although its creep resistance was quite good. Again, in the high-bismuth alloy, C-299, in comparison with the low-bismuth alloy, C-298, one sees the great reduction in ductility as time for failure is increased, although in the short-time tests this alloy has plenty of ductility.

I should like to point out that in Fig. 2 the arsenical alloys, C-389 and C-417, have fairly uniform ductility of 30 per cent or better through all the tests so far reported. Some of these tests have gone on now for over 16000 hr. That, however, is a very short time in the expected life of a cable.

The arsenical alloy C-389 has quite high ductility, except in the range around 100 hr. of tests. We find quite a few of these materials tend to decrease in ductility as the time for failure is increased, and then seem to increase in ductility for greater times for failure. The ductility depends upon the amount of slip within the grains and flow at the grain boundaries. Under favorable stress conditions, a maximum proportion of both slip and flow is obtained and likewise under certain stress conditions a minimum is obtained.

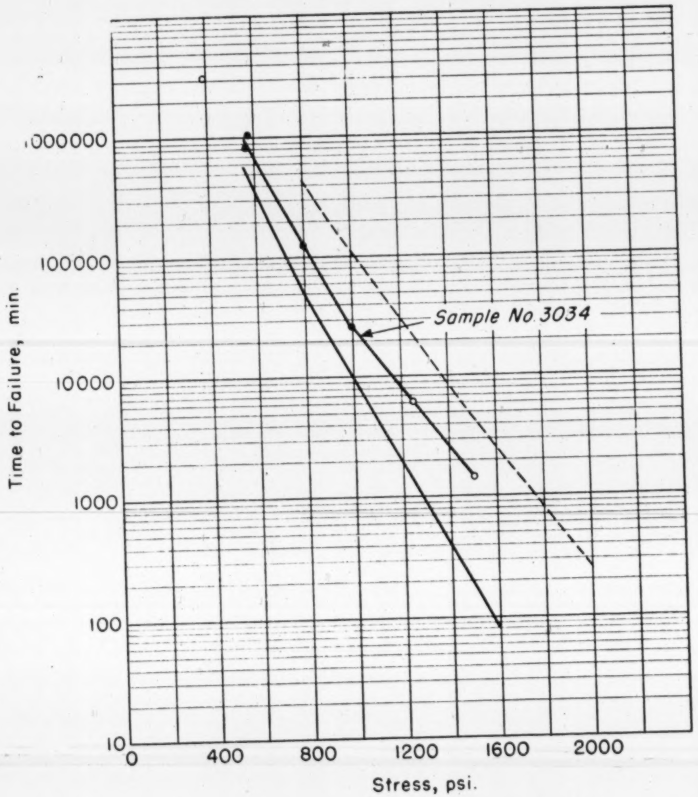


FIG. 3.—Comparison of Stress-Rupture Curves.

Reproduction of Phelps' Fig. 3, including only the two curves for sample No. 35 with Discusser's data added.

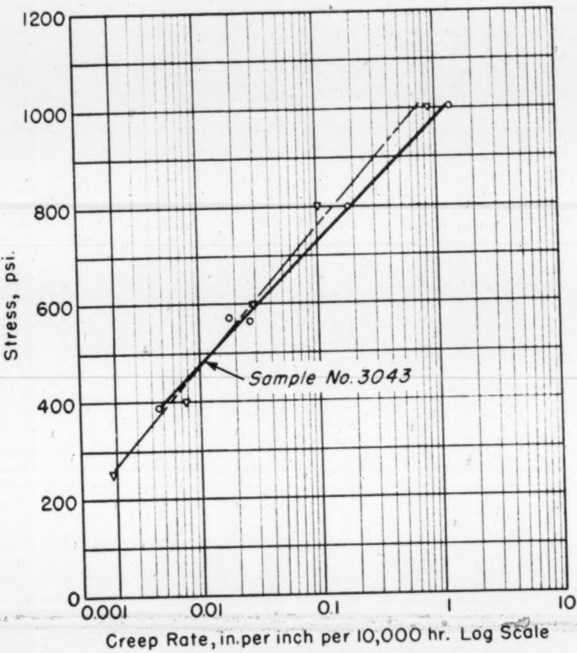


FIG. 4.—Relation Between Creep Rate and Stress.

Reproduction of Gohn's Fig. 8, including only the curve for alloy No. 35, with Discusser's data added.

MR. A. A. SMITH, JR.[2] (*presented in written form*).—The effect of silver on the creep of lead, as shown by the present authors, agrees quite well with the work done by Greenwood and his associates. It should be pointed out that creep rates at the lower stresses are of the most importance since cable sheath cannot expand to any great extent, as a useful life of 30 to 40 years is desired.

There is one question that I should like to raise and that is the effect of copper on an alloy containing no silver. I think that it is unfortunate that such an alloy was not included in the present studies because work done in this laboratory indicates that its creep properties will be the same regardless of silver content in the ranges studied by the present authors.

MR. C. J. SNYDER[3] (*presented in written form*).—Our laboratory has made many creep and stress-rupture tests, some of which were reported to this Society in 1940 by Bassett and Snyder under the name of burst tests.[4]

One sample not included in our paper was a high-silver copper-bearing lead somewhat similar to Phelps' sample No. 35. This sample (No. 3043) analyzed:

Silver, per cent	0.012
Copper, per cent	0.053
Bismuth, per cent	0.088
Antimony, per cent	0.005
Tin, per cent	0.00
Tellurium, per cent	0.005

Stress-rupture tests were done at stresses of 1500, 1250, 1000, 800, 576, and 566 psi. and a creep test at 391 psi., all at room temperature, and a creep test at a stress of 322 psi. and 110 F. These data are listed below:

STRESS-RUPTURE AND CREEP CHARACTERISTICS OF SAMPLE NO. 3043. (15 IN. LONG EXTRUDED TUBES TESTED AS DESCRIBED BY BASSETT AND SNYDER.[4])

Stress, (PD = 2 St), psi.	Pressure Medium	Total time, min.	Total Expansion or Creep, per cent	Creep Rate, Tangent rate (v), per cent per year	Type of Fracture
1500	oil	1 516	14.7		half brittle
1250	oil	6 218	10.1	...	half brittle
1000	oil	26 380	6.4	...	brittle
800	oil	135 530	4.4	...	brittle
576	water	1 156 800	3.70	1.7	brittle
566	oil	846 100	4.05	2.5	brittle
391	water	3 105 760	2.40	0.41	no failure
322	oil at 110 F.	108 120	0.093	0.40	no failure

When the stress-rupture data are plotted on Fig. 3 in Phelps' paper, the points fall about midway between the two curves for sample No. 35 (see accompanying Fig. 3). This is to be expected because the lower curve was plotted from tests at a higher temperature (110 F.) compared to our 75 F. The upper curve was plotted from tests reported in the Gohn and Ellis paper on strip specimens which usually take longer than tube specimens to break.

It should be noted, also, that the time to rupture is shorter when a lead tube is tested with oil pressure than when tested with water pressure.

When creep rates are calculated from our data and plotted on Fig. 8 of the Gohn and Ellis paper, the points fall rather close to their curve for alloy No. 35 (see accompanying Fig. 4).

It may be noted that we omit the calculation of creep rates for specimens tested at stresses at which the specimens failed in less than the stated period. We believe that such values are fictitious and lead to confusion.

Since our sample was composed of commercially pure lead, this similarity of test values indicates that the authors' samples of special lead were also representative of commercial lead.

---

[2] General Superintendent, American Smelting and Refining Co., Central Research Lab., Barber, N. J.

[3] Supervisor, Metallurgical Laboratory, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

[4] W. H. Bassett, Jr., and C. J. Snyder, "The Testing of Lead Cable Coverings," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 910 (1940).

We would like to see these authors indicate on Fig. 8 where chemical lead creep values would fall. Considering that the creep rate of chemical lead is quite well known, how do these rather pure silver alloys compare with it. Some engineers have attributed the comparatively good creep properties of chemical lead to its silver content, but we have always thought that the low creep was caused by the slight impurity of nickel present in most chemical lead. We added from 0.003 to 0.01 per cent nickel to two of the relatively less creep-resistant grades of commercial lead and several lead alloys and noted a big improvement. Nickel also had the power to overcome in some cases the adverse effects of bismuth and tellurium.

The discussion by Gohn and Ellis of brittle fractures *versus* stress was highly interesting. We have been studying this point for some time, as indicated in our 1940 paper. Some samples have been tested which did not give brittle fractures in creep tests at any stress. If the cause of the brittle fractures can be overcome, the stress-rupture life of many samples will be greatly increased. It is hoped there will be future reports of this work on this subject.

MR. HERMAN HALPERIN[5] (*presented in written form*).—These two papers give some very interesting data obtained at 400 psi. and above in the fracture tests and at 100 psi. and above in the creep tests. Messrs. Gohn and Ellis make a very important observation at the end of their paper in saying that, "Wherever possible, creep rates should be obtained for the stresses of interest from test data rather than by extrapolation from data taken at higher stress levels." Based on studies of detailed laboratory data and on studies of operating experiences and requirements, it seems to me that final judgment as to the expected characteristics of lead or lead alloys in service must be based on long-time tests at stresses in the general region of the operating stresses in service.

As many know, research on the properties of lead and lead alloys have been under way at the University of Illinois for over 20 years under the sponsorship of the Utilities Research Commission. Up to a few years ago, this work was under the direction of H. F. Moore, and since then has been under the direction of C. W. Dollins. As indicated in the bulletins issued by the researchers from the University of Illinois, including the one to be issued this summer, fracture tests are made at stresses down to 400 psi. These tests, just as do the tests described in the papers, have given important information relating to ductility. Then there have been tests on strips running up to 10,000 or 20,000 hr. or more, and tests on cylindrical samples of lead and lead alloys running from several years to as much as 13 years. These tests are at room temperature and at elevated temperatures of either 110 or 150 F. Furthermore, these tests have been at stresses of 50 to 250 psi. for ordinary leads and at stresses of 100 to 350 psi. for lead alloys. The power-cable specifications of the Association of Edison Illuminating Companies have set 125 psi. as the approximate limiting stress for ordinary commercial leads, including copper-bearing lead.

These long-time tests at the University of Illinois show that extrapolation of creep rates from results of tests of a few thousand hours to many years in service can be misleading, because frequently the rate of creep changes through tests running for a year or several years.

The creep tests have shown no

---

[5] Staff Engineer, Engineering Dept., Commonwealth Edison Co., Chicago, Ill.

particular advantage for lead containing a small amount of silver as compared to commercial leads including copper-bearing lead.

The tests, however, at and below 200 psi. at various temperatures show rates of creep for two arsenical lead alloys roughly one-tenth of the rates of creep found for copper-bearing lead. Furthermore, and quite importantly in the consideration of electric power cables, these arsenical lead alloys have lives to fracture in various bending tests of cables in Chicago and in tests of strips of sheath in University of Illinois apparatus that are about three to five times the lives of the various types of commercial lead. Sheathing containing some silver are included in the latter category. Also, as developed in both the short- and long-time creep tests and in bending tests on aged samples of cable, sheaths of these arsenical leads have elongations to fracture that are of the order of the elongations obtained for various commercial leads and do not develop hardness and brittleness with age—all in contrast with contrary findings of about 10 years ago with some calcium-type alloys.

MR. J. NEILL GREENWOOD[6] (by letter). —I would like to express an opinion on this work from two points of view (1) The utility of tests of this kind. (2) The results of the tests.

To my mind there are two criteria which can be applied to determine whether a series of tests is worth carrying out. In the first place will the results be of theoretical or technological interest and in the second place is the test material in a stable or at least a reproducible state? If results of theoretical value are expected, then extreme care is necessary with regard to the metallurgical condition of the test specimens. In the present work no claim is made that this criterion has been met, for the test specimens were milled from flattened sections of industrially extruded pipe. Even with pure lead this would give an indeterminate condition because of the cold deformation imposed, but in the case of silver alloys there is also the factor of the solid solubility of silver which varies from 0.1 per cent at 300 C. down to 0.01 per cent at ordinary temperatures. I suppose therefore that the major expectation from these tests was data of technological interest. In this connection I would feel doubtful, as a result of our work in Melbourne, of any correlation between results on test specimens prepared as described and results on the original extruded pipes because of the possible effects of the cold work done in flattening the pipe. The creep rate of lead and its alloys is very sensitive to residual effects from cold working.

In a recent communication[7] we have confirmed what others have also found, that with "pure" lead there is a transition in the relationship between rate of creep and applied stress. This transition occurs at about 300 psi. and indicates that above this a change in mechanism of deformation occurs. We have called this phenomenon the "creep yield" because of the similarity to the yield phenomenon in ordinary tension testing. In practice no material would be used above this stress and it would seem that it is a first point to determine for any metal or alloy. Data obtained from stresses above this bear no relationship to the behavior of the given metal under practical conditions and therefore could be eliminated from technological testing. Stresses of the order of those met in practice necessitate more accurate strain readings than those with stresses above the creep yield. Also, our experience leads us to the conclusion that with lead and its alloys, tests of less than one year's duration are apt to be misleading, and it is preferable to use stress duration of several years. Hence the useful part of the present results in so far as they are of technological interest lies in the field below 400 psi. stress and over 10,000 hr. duration. For future work it would be considered desirable to determine the creep yield stress first by making accurate strain measurements over a period of, say, several weeks and then to start the main program. So far as we know, calcium is the most effective element in raising the creep yield stress.

---

[6] Research Professor of Metallurgy, The Baillieu Laboratory, University of Melbourne, Victoria, Australia.

[7] *Metallurgia*, April, 1948.

Turning now to the actual results, the most interesting point to me is the statement in the conclusions that the effect of silver is greater above a stress of 400 psi. than below. If the percentage elongation in 16,000 hr. is plotted against stress, it will be seen that a marked increase in rate of creep occurs at stresses over 400 psi. This, then, is approximately the creep yield stress (except for the basis lead where a value of 300 psi. is found) for this group of materials. The number of points given does not admit of closer evaluation.

Silver in the amounts used in these alloys would be mostly in solid solution (though this is an uncontrolled variable) and would therefore be expected to decrease the creep rate for those stresses which cause a preponderance of movement within the crystals. My interpretation of the "creep yield" stress is tentatively that below this stress creep is essentially noncrystalline (viscous) possibly associated with grain boundaries whilst above this it is associated, in a manner not yet determined, with the body of the crystal. If this is the case, it would be expected that the influence of silver would be greater (in reducing the creep rate) at stresses above the creep yield stress, that is, above 400 psi.

The technological importance of the creep yield stress from the point of view of creep testing is that only tests carried out below this stress are significant because they involve the same type of deformation as that which occurs under engineering conditions. It also explains why rapid tests carried out at high stresses are useless for evaluating the creep resistance of materials.

In a series of tests with silver-lead alloys having 0.03 to 0.10 per cent silver—specimens rolled and annealed 15 min. at 100 C.—the following results were obtained under stress of 350 psi. at 20 C.

Silver, per cent	Elongation, per cent				
	1 yr.	2 yr.	3 yr.	4 yr.	5 yr.
0.0003	2.2	3.8	5.0	6.2	7.2
0.03	1.0	1.9	2.8	3.6	4.4
0.05	1.5	3.0	4.4	5.8	7.4
0.10	1.8	3.9	5.7	7.6	9.5

It will be noted that these rates of creep are substantially higher than those recorded by the authors. Further, it will be noted that only the 0.03 per cent silver alloy shows a lower creep rate than that of the basis lead. In all of these alloys some of the silver is present as a dispersed phase.

In the case of copper additions, it has been shown that the effect depends on the degree of dispersion of the copper particles. The following results, comparable with those above have been obtained.

Copper, per cent	Elongation, per cent				
	1 yr.	2 yr.	3 yr.	4 yr.	5 yr.
0.0002	2.2	3.8	5.0	6.2	7.2
0.055	1.6	2.8	3.7	4.4	5.2
0.066	0.1	0.2	0.4	0.5	0.6

It should be pointed out that these were rolled from small laboratory ingots and it has been shown that with more coarsely dispersed copper particles there is no advantage in adding copper.

Some results have also been obtained on two industrial lead sheets containing 0.01 per cent silver and 0.01 per cent silver with 0.06 per cent copper. These were tested under a stress of 350 psi. at 50 C. (122 F.). The results on Port Pirie lead under the same conditions are given for comparison.

Silver, per cent	Copper, per cent	Elongation After 300 days, per cent
0.0003	0.0002	6.2
0.01	0.0002	11.8
0.01	0.06	3.2

Our general conclusion, which is substantiated by the authors', is that silver and copper are not worth-while additions to lead from the point of view of improvement of creep resistance.

MESSRS. G. R. GOHN AND W. C. ELLIS (*authors' closure by letter*).—The importance of obtaining creep data on lead cable sheathing alloys in the stress range of 100 to 300 psi.—the range of engineering interest—has been further emphasized by Mr. Dollins. Such tests can be made with instrumentation now available, but it should be recognized that at these low stresses the tests must be continued for long periods of time before reliable creep rates are established. The important thing in planning such tests is that the tests for establishing creep rates be conducted in the stress range for which the mechanism of creep is essentially the same as that occurring at the stress of practical interest. This we have done in the case of those tests conducted at stresses of 400 psi. and below.

On the other hand, it is equally essential to know how the ductility of the sheathing alloy changes with stress level while the sheath is subjected to continuous creep loading. This can only be accomplished by high-stress short-time stress-rupture tests. Thus in our own work, the stress rupture data show the undesirability of increasing the silver content beyond 0.010 per cent without an increase in copper content to offset the embrittling action of the higher silver content. However, as pointed out by Mr. Dollins, the use of these high-stress creep rupture tests to evaluate the creep resistance of lead sheathing alloys at lower stresses may definitely lead to erroneous impressions.

The additional data offered by Mr. Dollins to show the effect of a slight increase in one constituent is welcome. We believe that investigations in this field—namely, the study of the effect of small additions, normally considered to be well within the impurity range, upon the chemical and physical properties of the resulting alloys—will repay any efforts spent in such studies.

The observations of Mr. Dollins with respect to the arsenical and calcium leads are interesting. However, it should be emphasized that calcium leads (alloy C-297) referred to in his discussion contained 0.016 per cent magnesium, a quantity sufficient to have a deleterious effect on the ductility. The binary, magnesium-free calcium-lead alloys compare quite favorably in their creep characteristics with the arsenical alloy, C-389.

We have no data which permit us to answer Mr. Smith's inquiry as to the effect of copper alone on the creep properties of lead. As pointed out by the authors, a previous paper (by Gohn, Arnold, and Bouton (**6**)[8] had indicated, the superior creep resistance of chemical lead containing 0.005 per cent silver and 0.05 to 0.06 per cent copper as compared to high-purity lead. Our present studies evaluate only the effect of variations in silver content upon the creep characteristic of lead when the copper content is held constant at 0.004 per cent and the beneficial effect of increasing the copper content to a value equal to approximately that found in chemical lead when the silver content exceeds 0.010 per cent.

---

[8] The boldface number refers to the list of references appended to the paper by Gohn and Ellis, see p. 814.

In reply to Mr. Snyder's discussion, the expression of creep rates on the basis of 10,000 hr. elapsed time is merely a matter of convenience. Where such values are fictitious, they may be reduced to real values simply by dividing the rate and time values by 10, 100, or 1000 as indicated by the data in Tables II to VII, inclusive, to insure that the elapsed time falls within the time marking the end of the second stage of creep.

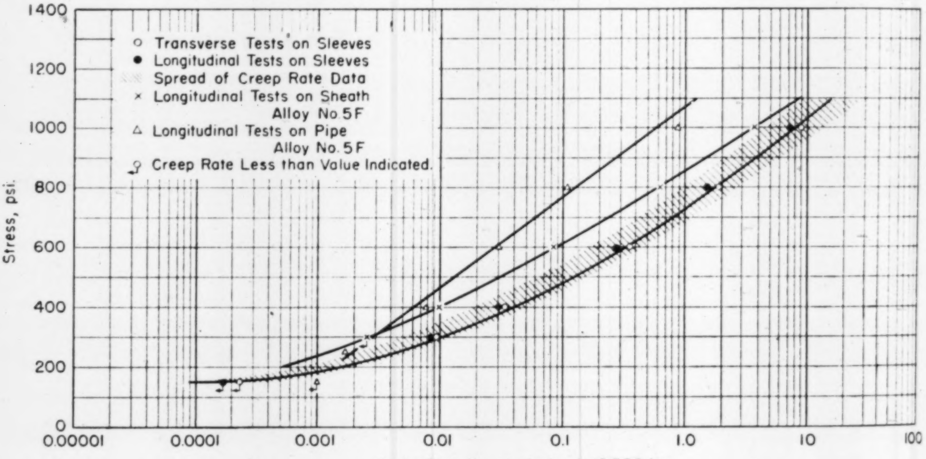


FIG. 5.—Comparison of Minimum Creep Rates for Chemical Lead Sheath and Sleeves with Alloy No. 35.

Mr. Snyder's request for a comparison of these data with those of chemical lead is timely and important. The accompanying Fig. 5 shows for purposes of comparison the creep rates of strip tension specimens of alloy No. 35 taken from Fig. 8 of the present paper, chemical lead cable sheathing alloy No. 5F extruded and aged $8\frac{1}{3}$ years prior to test, and chemical lead sleeves. The data on the sleeving alloys represent duplicate tests on two sleeves from each of two producers. Two transverse test specimens were taken from each of four sleeves, but only one longitudinal specimen was tested from each of the same sleeves so that the scatter band shown in Fig. 5 represents the spread for 12 creep tests on four different sleeves, half from one supplier, half from another. The close agreement of these data should be noted.

Mr. Halperin's comments offer valuable additions to our paper. They further emphasize the need for making creep tests at engineering stresses rather than to predict creep behavior from stress-rupture data.

Mr. Greenwood has discussed a number of the important points of this paper. It is gratifying to have in discussion the benefit of his extensive experience, representing as it does, a long series of valuable investigations and contributions to our knowledge of the creep of lead and its alloys.

The effect of the metallurgical condition of the test specimen on the creep results obtained is worthy of consideration in initiating a series of tests. We believe that specimens used to obtain technological creep data, for sheath applications, which was the primary purpose of this paper, should initially be in a condition as nearly like the condition of use as is practically possible. We believe that this was achieved by using specimens from material prepared by extrusion. The flattening of the tension test specimen does not appear to us to be a factor which diminishes the value of the results for the following reason. In practice, sheath is reeled and unreeled prior to installation and therefore goes into service with a prior history of plastic deformation. It turns out that this plastic flow is comparable to that resulting from the flattening of a tension test specimen.

As a matter of interest, the flattening of the tension test specimens resulted in no microscopically detectable difference in the etched structure. Recrystallization did not occur as a result of the straining, at least in several months after flattening which covered the period of examination.

There is no question but that prior strain will affect creep behavior pronouncedly in the initial period of the test and possibly to a lesser extent in the later stages of test. The conditioning of the specimens used, however, was in the direction of the service conditioning and the results therefore reflect behavior under service conditions.

We agree that the structural condition of a lead alloy as respects the presence and distribution of phases has a marked effect on creep rates. The behavior of lead-antimony alloys is a noteworthy example. In alloys such as lead-silver, structural changes have freedom to occur throughout the creep test and very likely influence the test results. The initial condition for test, however, is within the control of the experiment. The selection of the extruded condition for test specimens most nearly meets the condition of material used in service for sheathing.

Mr. Greenwood in his statement that tests carried out at high stress levels are useless for evaluating the creep resistance of lead alloys was undoubtedly further emphasizing the dangers of extrapolating creep rate behavior at high stress levels to the lower stresses of practical importances. This has been amply demonstrated by the results in the Gohn and Ellis paper, Fig. 9, and in previous papers (**6**). In the broader concept of creep resistance the inclusion of high stress data where failure occurs is, however, important. Such tests show the susceptibility of the material to loss of elongation or embrittlement as the stress level is decreased or as the time to failure is increased. This is illustrated in Fig. 5 of the Gohn and Ellis paper in which a marked falling off in ductility was found for the alloy of highest silver content at 600 psi. The value of elongation-at-fracture measurements is also emphasized in Mr. Dollin's discussion. A low elongation—evidence of brittleness—may not prevail at low stresses even though present at intermediate stresses, but there is not likely to be an answer to this in the immediate future. Lacking specific data for brittleness susceptibility at low stresses we consider that a marked falling off of the ductility in the intermediate stress levels for a lead alloy makes the practical use of the alloy questionable.

We are in agreement with Mr. Greenwood that there is considerable evidence indicating that the mechanism of flow is different at high stress levels from that operating at low stresses. Direct experimental evidence of a microscopic or equivalent nature would be welcome to confirm what is at present only an explanation of observed property relationships.

The data supplied for a series of lead-silver, lead-copper and lead-silver-copper alloys in a different initial condition and in one case at a high testing temperature are a welcome addition to the data presented in the Gohn and Ellis paper.

MESSRS. GOHN AND ELLIS (*presented in written form*).—There are many interesting points in this valuable contribution of Messrs Phelps, Kahn and Magee to the knowledge of the creep of lead and its alloys. We wish to discuss two of these.

From Fig. 2 of their paper it is evident that the time-to-failure in strip-tension specimens is, in most instances, substantially longer than for pipe specimens at the same calculated initial stress and temperature. This finding deserves study, for by itself it appears to show that strip-tension tests give optimistic results for the life of sheathing.

The two methods of test differ in important respects which lead to an uncertainty that the stresses are initially the same in a given comparison of life or that the instantaneous stresses existent at any time in the course of the test have the same values even though they may initially have been the same. The presence of an uncertainity in the initial stress has already been pointed out by the authors in their comment on a triaxial stress system in the case of the pipe.

If we consider the instantaneous values of stress which exist in the strip-tension specimen at any time there must be an increase in stress due to the decrease in cross-section, as the specimen elongates. For the pipe sample the increase in stress is due both to a cross-sectional decrease and to the increase in diameter of the pipe which effectively increases the load on the stressed cross-section. Phelps, Kahn and Magee have previously discussed this viewpoint in an earlier paper.[9] Qualitatively, without going further into the analysis, this would seem to indicate a more rapid increase in stress, particularly in the late stages of life, for the pipe samples and hence early failure as was observed. This difference would be minimized for small elongations and, if useful life were considered to be limited to 3 to 5 per cent elongation, not much difference in time would be required in the two methods to reach this range of values. This essentially considers the problem as one of creep rates rather than one of time-to-failure which, to the present discussors, appears to have a definite advantage for obtaining design information. Considerable value would exist in a comparison of creep rates in the range of small elongations for the two methods of test. We are now therefore initiating a series of tests to obtain information on this correlation.

The calculation of initial stress in the pipe samples is a difficult one in view of the limited elasticity of lead. The formula given by the Phelps, Kahn and Magee results in a calculated stress approximately 10 per cent less than that resulting from the use of the thin shell theory. As a matter of interest, the discussors replotted the data of Fig. 2 using stresses for the pipe specimens as calculated from the thin shell theory and obtained as good agreement in life between the two methods as might be expected. A similar agreement was obtained on other lead alloys in an earlier investigation by Moore, Dollins, and Craig.[10] This agreement may be fortuitous but as experimental evidence it indicates that, when strip-tension data are used for establishing the life of pipe, the thin shell theory should be used for design.

---

[9] Howard S. Phelps, Albert M. Gates, and Frank Kahn, "Internal Hydraulic Bursting Tests of Lead Cable Sheathing," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 885 (1940).

The data in Fig. 6 showing the effect of temperature on creep range for this series of alloys are a valuable and substantial addition to the small amount of information in the literature on this subject. The temperature dependence of creep rate is of practical importance and theoretically useful in connection with the study of the mechanisms involved in creep.

MR. GEORGE O. HIERS.[11]—In the paper by Phelps, Kahn, and Magee it is stated that sodium improves the chemical composition stability. I wonder whether we could obtain a little more enlightenment thereon? Does the inprovement pertain to deoxidation upon remelting or to homogenization?

MESSRS. HOWARD S. PHELPS, FRANK KAHN, AND WILLIAM P. MAGEE (*authors' closure by letter*).—Comparison of specifications for the A.S.T.M. grades of lead indicates there is a substantial overlap in the percentages of minor constituents present. It is thus possible there may be little or no difference in chemical composition between two samples of lead presumably of different grades. For instance, a particular sample of lead may meet the requirements for both chemical and copper lead. Similarly, a composition might be selected which would meet the requirements for both copper and acid lead. This has led to considerable confusion and emphasizes the importance of knowing the actual percentages of these minor constituents present in particular samples rather than have them identified only by A.S.T.M. grade.

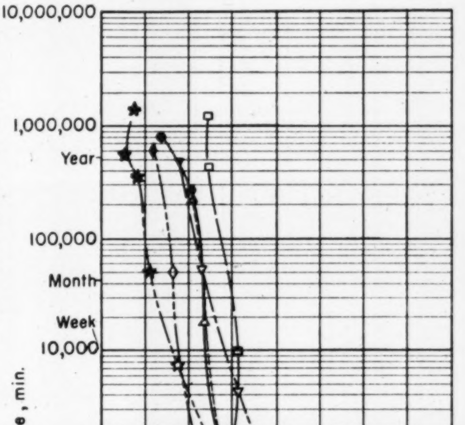


FIG. 6.—Ductility-Time Curves for Bursting Tests of Lead Sheathing Pipe at 110 F.

Open characters indicate necked-down failures. Solid characters indicate radial failures. The letter I denotes intercrystalline failure and W weld failure.

A large number of bursting tests on cylindrical specimens were made by the present authors on various samples of commercial lead sheathing, comprising several A.S.T.M. grades. These tests showed there was no consistent correlation between the grades of lead and the stress-rupture characteristics that were observed. In establishing current A.S.T.M. grades in terms of content of minor chemical constituents, it would appear that the influence of these constituents on the stress-rupture characteristics had been disregarded. Further investigation of the influence of these minor constituents on the stress-rupture characteristics seemed warranted and led to the present tests on a series of samples carefully prepared to vary a single constituent, namely, silver, which the evidence in hand indicated might be a controlling factor.

---

[10] H. F. Moore, C. W. Dollins, and W. J. Craig, "Bursting Tests and Tension Tests for Lead Cable Sheathing," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 904 (1940).

[11] Metallurgist, National Lead Co., Research Labs., Brooklyn, N. Y.

It is of interest to note how the slight differences in chemical composition of the authors' samples change their A.S.T.M. grade classification. Sample 30 meets the chemical requirements of all specifications B 29 grades of lead except chemical lead, acid lead, and copper lead. Sample 31 meets the requirements only of soft undesilverized lead. Samples 32, 33, and 34 do not meet the requirements of any of the A.S.T.M. grades. Sample 35 meets the chemical requirements only of chemical lead and copper lead.

For purposes of comparing results of tests for effect of chemical composition, it seems essential that the actual chemical analysis of a sample be specified rather than merely stating its classification as an A.S.T.M. grade.

Mr. Dollins states that a report of the ductility (maximum increase in diameter) of the cylindrical sheath samples would be very desirable. Such data are shown in the ductility-time curves of the accompanying Fig. 6, in terms of time-to-failure *versus* maximum increase in diameter. These curves indicate that at operating stresses, for all except samples 31 and 32, the maximum increase in diameter will be of the order of 5 per cent or less. Data are not yet available for samples 31 and 32 at stresses below the equicohesive stress, but there is little doubt they will follow the same trend. It is important to note that all of these compositions show decreasing ductility with decrease in stress, as contrasted with strip-tension test results shown for some of the compositions of Mr. Dollins' Fig. 2, which indicates increasing ductility with decreasing stress.

In the work by the authors on cylindrical specimens, it is to be understood that, due to eccentricity of the specimens, the stretch was not uniform around the circumference. However, the samples were all of comparable low eccentricity. The average coefficient of eccentricity (ratio of minimum to average thickness) was 0.965 with maximum deviation of ± 0.010. The data are therefore believed to be reasonably representative of the relative performance of the several compositions.

Excessive expansion, such as might occur with a lead having high ductility at low stress, is an undesirable characteristic for cable sheathing. For example, a sheath which would eventually expand 25 per cent or more in diameter at operating stresses would have an increased core volume of 56 per cent with the accompanying maintenance problem of assuring positive oil pressures. Moreover, the lead thickness would be decreased 20 per cent, with a corresponding increase in stress for the same internal pressure, thereby accelerating expansion and ultimate failure.

On the other hand, an increase in diameter of 5 per cent or less would entail a volumetric increase of not over 10 per cent and an increase in stress of

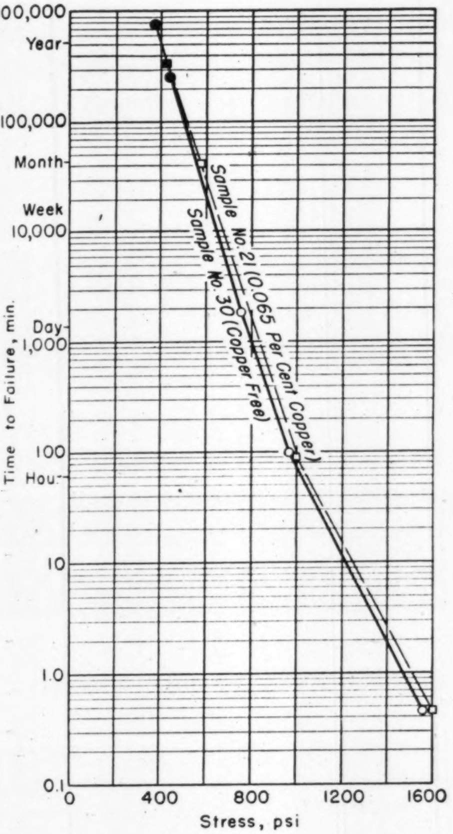


FIG. 7.—Comparison of Stress-Rupture Curves for Bursting Tests of Lead Sheathing Pipe at 110 F.

Open characters indicate necked-down failures. Solid characters indicate intercrystalline failures.

less than 5 per cent. Deformations of this magnitude can be readily tolerated in practice.

The request of Mr. Smith for information on the effect of copper on an alloy containing no silver is timely. Some data on this point, available from stress-rupture tests at Philadelphia Electric Co., are shown in the accompanying Figs. 7 and 8.

A sheathing pipe sample of high purity electrolytic lead to which 0.065 per cent copper had been added was received from a cable manufacturer who supplied the following chemical analysis made on an adjacent section:

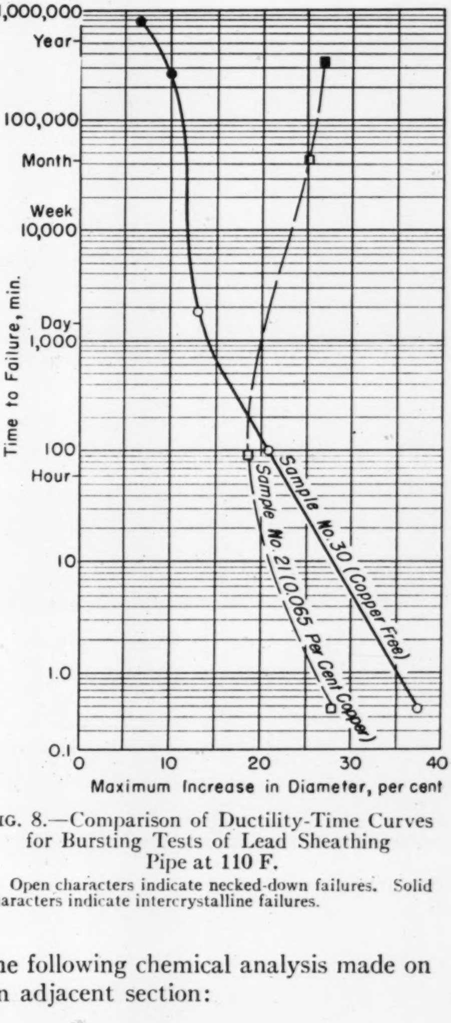


FIG. 8.—Comparison of Ductility-Time Curves for Bursting Tests of Lead Sheathing Pipe at 110 F.

Open characters indicate necked-down failures. Solid characters indicate intercrystalline failures.

Chemical Composition Exclusive of Lead

Bismuth, per cent	0.0005	
Copper, per cent	0.065	
Silver, per cent	0.0002	
Antimony, per cent	0.002	(less than)
Tin, per cent	0.0001	(less than)
Nickel, per cent	0.0005	(less than)
Iron, per cent	0.0002	
Tellurium, per cent	0.001	

This sheathing pipe is identified as sample 21. Except for the small percentage of tellurium present, this sample would appear to meet the chemical requirements of the acid lead or copper lead A.S.T.M. grades.

Figure 7 compares the stress-rupture curve for sample 21 with that for the high purity lead, sample 30. These curves are substantially identical, indicating little if any effect of the presence of 0.065 per cent copper on the stress-rupture characteristic.

Figure 8 compares ductility-time curves for samples 21 and 30. This shows that this copper content (0.065 per cent) has a very marked influence on ductility, giving a 27 per cent increase in diameter as compared with approximately 10 per cent increase in diameter for the copper-free sample at about the 400 psi. stress level.

Considering only ability to withstand internal pressure, the copper-free lead would seem preferable for use as power cable sheathing. The addition of copper alone, to pure lead, apparently produces the undesirable characteristic of increased ductility without increase in life.

In reply to George O. Hiers' request for information on the sodium treatment for stabilization of lead, he is referred to a paper by B. B. Reinitz and R. J. Wiseman, entitled, "A New Technique for Lead Cable Sheathing," published in the *Transactions* of the American Institute of Electrical Engineers, Vol. 59, 1940, March Section, pp. 165–176. According to these authors, metallic sodium introduced into the melt combines with any oxygen present and decomposes and throws out of solution deleterious compounds frequently present in commercial lead. The residual sodium in the treated lead is said to be of the order of 0.001 per cent.

Messrs. Gohn and Ellis are to be commended on their well presented companion paper reporting their thorough and painstaking investigation of the creep characteristics of these interesting silver-bearing lead alloys. It is hoped their creep data together with the authors' stress-rupture data on the same samples may prove helpful in leading to an understanding of changes in the fundamental physical properties that result from incremental additions of silver to lead.

CHANGES FOUND ON RECIPROCATED STEEL, CHROMIUM PLATE, AND CAST IRON SLIDING SURFACES*

By J. N. Good[1] and Douglas Godfrey[1]

In order to obtain a better understanding of the phenomena of wear and wear resistance, the changes that occur due to rubbing metallic surfaces on one another have been studied by many investigators. A study of the appended references reveals that at least the following factors are of importance:

1. Formation of surface films and reaction products,
2. Changes in surface hardness, and
3. Changes in crystallite size.

Some relations are established herein among these factors and the phenomena of run-in and one phase of wear—scuffing—by identification of and examination of surface changes occurring during one of the well-known sources of metallic wear, reciprocating sliding. The term "run-in" is used in this paper to designate the process through which surfaces acquire, by mutual interaction during sliding contact, increased capacity to carry load and to withstand wear. This investigation neglects the relation of hydrodynamic and boundary lubrication to run-in, although all surfaces studied were lubricated.

Changes in the surface composition and surface characteristics were studied under run-in and wear conditions for forged steel (S.A.E. 4140) rubbed against cast iron, chromium-plated cast iron rubbed against steel, and cast iron rubbed against cast iron; these combinations are standard in aircraft engines. Use was made in this investigation of specialized techniques in X-ray and electron diffraction, of chemical and metallographic analyses, and of hardness measurements.

Apparatus and Procedure

Specimens of standard aircraft-engine cylinder-barrel and piston-ring material combinations used in these examinations were obtained from two sources. Portions of used cylinder barrels and piston rings were directly examined, whereas portions of unused cylinder barrels and piston rings were examined after rubbing under pressure in a reciprocating slider machine. Surface changes of the specimens were examined by X-ray and electron diffraction. Supplementary information was obtained by chemical and microchemical analysis, electrographic analysis, metallographic examination, and microhardness determination.

The following materials were investigated:

Material Combination	Specimen Examined	Specimen Source
S.A.E. 4140 steel slider rubbed against cast-iron rider	Slider	Slider machine
Cast-iron piston rings rubbed against S.A.E. 4140 steel cylinder barrel	Cylinder barrel	Aircraft engine
S.A.E. 4140 steel slider rubbed against chromium-plated cast-iron rider	Rider	Slider machine
Cast-iron slider rubbed against cast-iron rider	Slider	Slider machine
Chromium-plated cast-iron piston rings rubbed against S.A.E 4140 steel cylinder barrel	Ring	Aircraft engine

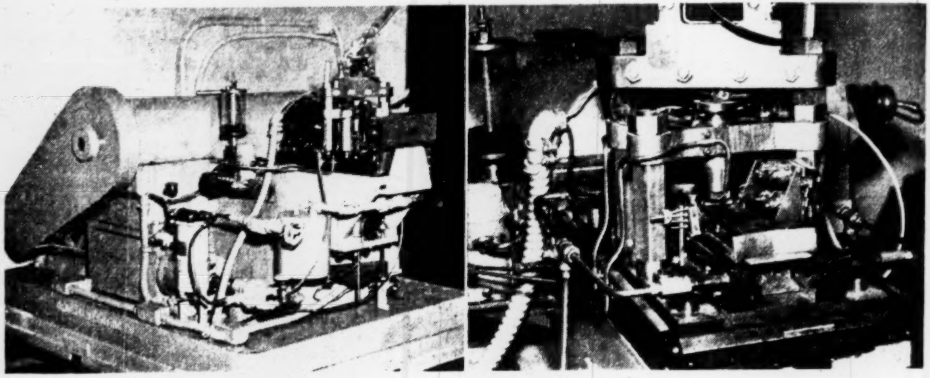
---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Flight Propulsion Research Lab., National Advisory Committee for Aeronautics, Cleveland, Ohio.

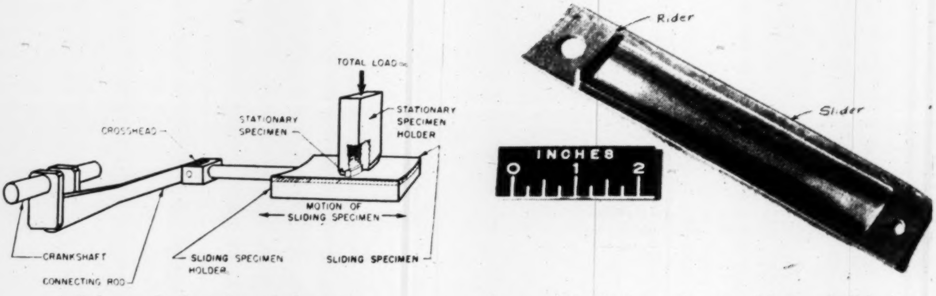
This selection of specimens is representative of the materials used in standard aircraft engines and should give results representative of run-in and scuff in those engines.

The slider machine (Fig. 1) reciprocated a slider specimen $7\frac{1}{2}$ by $1\frac{1}{2}$ in. beneath a fixed and loaded rider $1\frac{1}{8}$ by $\frac{1}{8}$ in. The rider was loaded by applied gas pressure, which was read from a dial. Mass temperatures of the rider and the slider were recorded with thermocouples. When the surfaces are lubricated, as in this investigation, conditions of both boundary and hydrodynamic lubrication are believed to exist, that of boundary at the ends of the stroke and that of hydrodynamic at the middle of the stroke. The oil used was Navy 1120 fed at a rate of 16 ml. per hour. The reciprocating speed was 630 cycles per minute with a stroke of $5\frac{1}{2}$ in. The procedure used for all specimens was as follows:



General view.

Detail of rider-loading mechanism.


TOTAL LOAD
STATIONARY SPECIMEN
STATIONARY SPECIMEN HOLDER
CROSSHEAD
MOTION OF SLIDING SPECIMEN
CRANKSHAFT
SLIDING SPECIMEN HOLDER
SLIDING SPECIMEN
CONNECTING ROD
Rider
Slider
INCHES
0
1
2

Schematic drawing of elements.

Typical specimens: rider and slider.

FIG. 1.—Reciprocating Slider Machine.

1. The two mating surfaces were lapped with silicon carbide lapping compound with resultant surface-roughness readings of approximately 40 to 60 microinches rms. for the rider and below 15 microinches rms. for the slider.
2. The surfaces were cleaned with solvents.
3. The surfaces were mated by recipro-

cation under a constant load of 600 psi. applied to the rider for 15 min. Run-in began at this point.

4. For scuffing specimens, the rider was loaded in increments of 100 psi. every 5 min. until scuffing occurred. A prolonged run-in procedure, consisting of operation at 2400 psi. for 2 hr. followed by 100 psi. and temperature-stabilization increments until scuffing occurred, was used on one cast-iron surface. Examples of the run-in curves for these procedures are given in Fig. 2. Scuffing observed visually, accompanied by rapid temperature rise, served as a criterion of surface failure. The machine was stopped within a few seconds after failure began, which was usually 2 to 3 hr. after the start of the run.

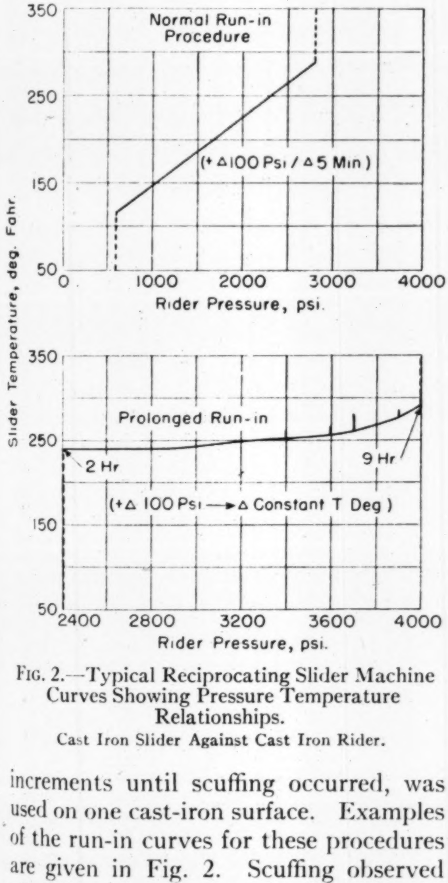


FIG. 2.—Typical Reciprocating Slider Machine Curves Showing Pressure Temperature Relationships.
Cast Iron Slider Against Cast Iron Rider.

The aircraft engines were operated for $5\frac{1}{2}$ to 7 hr. of run-in plus 15 to 100 hr. of endurance at an average engine speed of 2500 rpm. and an average indicated mean effective pressure of 250 psi.

All X-ray diffraction patterns were obtained with cobalt K_α radiation and with the specimen mounted in the manner used with the Debye-Scherrer powder camera. A 5-in. diameter cylindrical camera was used with an iron-oxide filter. Diffraction-line intensities were visually estimated. A microphotometer could be used only to record α-iron X-ray-line position, breadth, and intensity for crystallite-size determination. Positive identification of compounds was made only when a minimum of three of the strongest lines was observed for each compound associated with at least one surface condition out of a series of surface conditions obtained for a given material. Calculations of crystallite size were made from line broadening according to the procedure used by Barrett **(1)**[2] on the assumption that crystallite-size effects alone were causing line broadening. Fine copper powder was used to determine the standard line breadth. No estimation of the fraction of this broadening caused by lattice distortion was made. (Lattice distortion may have caused a great part of the broadening but no proved way exists to measure relative effects of lattice distortion and crystallite size.) If, in a group of lines composed of diffraction patterns of several compounds, one pattern obviously predominated in intensity, that compound was believed to be present in greater amounts than any of the others.

Electron diffraction examination of subsurface regions in the used engine specimens was made after each of five successive abrasions of the metal on 00 emery paper under pure benzene. The depth of an abrasion was measured with a vernier micrometer. Estimates of the depth of surface changes were made by microscopic measurements on cross and taper sections. Control specimens cut from the base structure of the engine cylinders were similarly treated and examined for evidence of any oxidation that might have been produced by abrasion.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 852.

Hardness measurements were made with either Knoop or Vickers indenters depending upon the condition of the surface being investigated. The quantitative units thus obtained were checked qualitatively with a Bierbaum scratch (microcharacter) hardness tester. This scratch indentation is less affected by metallic strata underlying the surface than the other indenters, but it is more sensitive to surface irregularities.

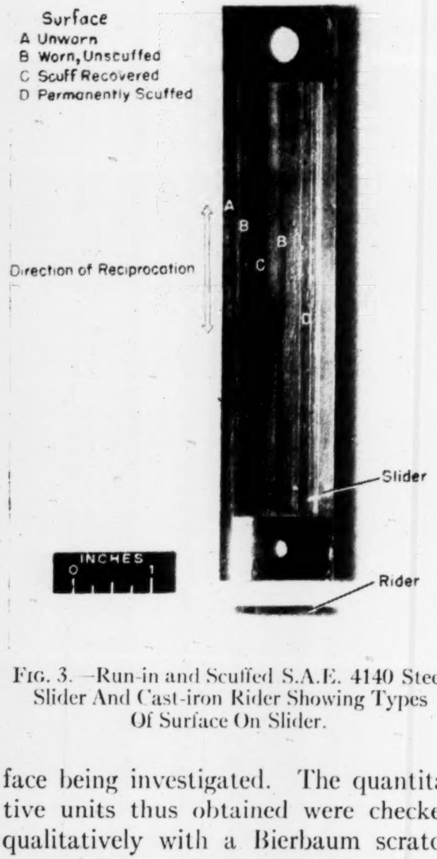


FIG. 3.—Run-in and Scuffed S.A.E. 4140 Steel Slider And Cast-iron Rider Showing Types Of Surface On Slider.

Chemical and microchemical analyses were made on natural and metallographic surfaces by means of solution, electrographic, and electrolytic etches. Metallographic examinations were made on normal or on tapered and plating-reinforced cross-sections.

RESULTS AND DISCUSSION

Steel Surfaces:

With the slider-rider combination of S.A.E. 4140 steel and cast iron, several conditions occurred successively in strips, or zones, on the steel slider (Fig. 3). The production of more than one surface condition on one specimen was due to slightly imperfect mating of rider and slider. The manner in which surface changes progressed represents various steps in increased loading until failure. Identification of the surface conditions is as follows: *A* represents the original unworn (unrun) surface; *B* represents a worn but unscuffed surface; and *C* represents a surface that appeared to have entered an initial condition of scuff rather early in the experiment but to have almost immediately recovered. Close observation during the run revealed a momentary darkening in color and then a change to dark gray. The permanently scuffed surface that developed in several stages, which succeeded each other within a few seconds, is represented by *D*. The first stage of formation was indicated by the momentary appearance of a dark-brown or reddish-brown surface color, which changed to black. The change in color was accompanied by the appearance in the recovered oil of a black sediment. The appearance of bright spots and heavy scuff marks on the surface accompanied by a sharp increase in temperature indicated permanent scuff. It appears that because of the initial imperfect mating a shifting of load took place, which led to variations and irregularities in local load concentrations. Surface *B* may be considered to have carried a large portion of the load during the mating procedure and therefore to have been able to support the load as it was increased. As the load was increased, surface *C* suddenly appeared. This appearance occurred as a result, possibly, of slight warping as the temperature rose and because surface *C* may have been insufficiently mated at the time the load increased. As the load was further increased, strip *D* appeared, possibly because of a combination of higher load and further warping until the surface scuffed rapidly with so much progressive damage that the scuff became extensive and permanent.

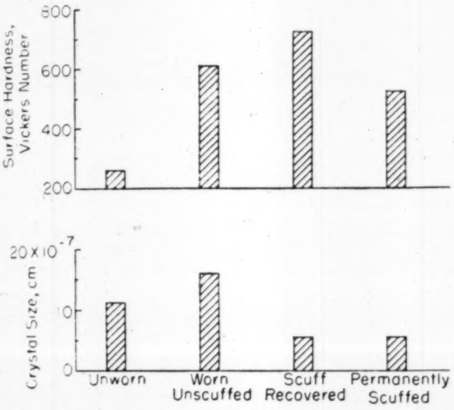


FIG. 4.—Relation Between Surface Changes and Surface Condition Of S.A.E. 4140 Steel Slider Specimens.

Identification of alpha iron (α-Fe), ferrosoferric (ferrosic) oxide (Fe_3O_4), and iron carbide (Fe_3C) in these surfaces was made by X-ray diffraction. (Whenever

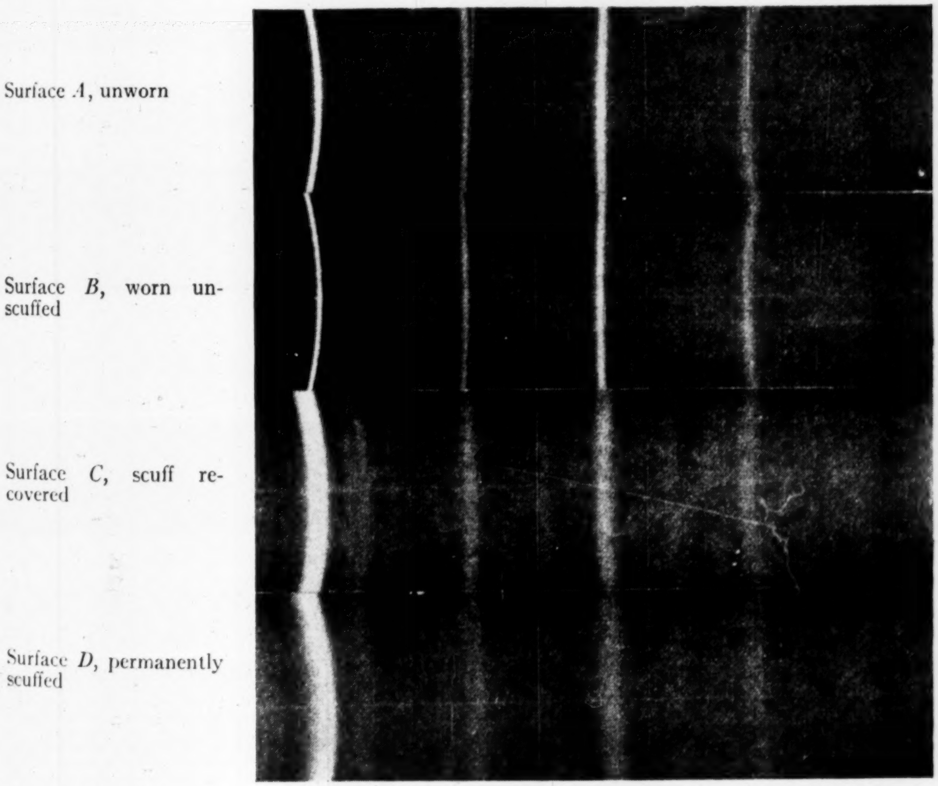
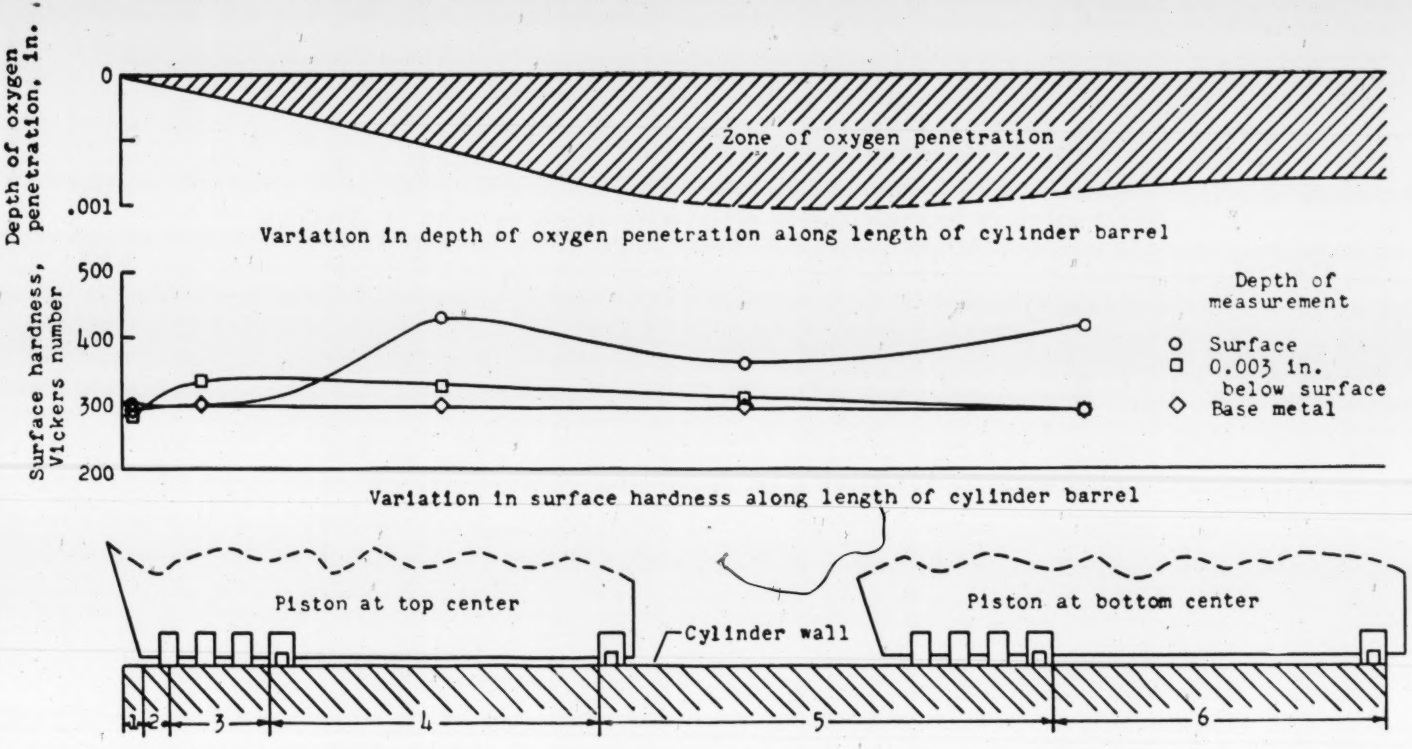

Surface *A*, unworn
Surface *B*, worn unscuffed
Surface *C*, scuff recovered
Surface *D*, permanently scuffed

FIG. 5.—X-ray Diffraction Patterns From S.A.E. 4140 Steel Slider Specimen Showing Change in Crystal Size From Surfaces *A* to *D*.



Area number	Location of area on cylinder wall with reference to piston	Piston position	Surface condition
1	Above piston travel	-------------	Thin oxide film, no wear
2	Opposite top land	Top center	Light pitting
3	Opposite compression rings	Top center	Heavier pitting and wear
4	Opposite skirt (high sliding speeds)	Top center	Light pitting
5	Opposite compression rings	Bottom center	Low wear
6	Opposite skirt	Bottom center	Very low wear

FIG. 6.—Relations Among Surface Condition, Material Hardness, and Depth Of Oxygen Penetration Found On Worn, Unscuffed S.A.E. 4140 Steel Aircraft Engine Cylinder Barrel.

Fe_3O_4 is mentioned herein, the possibility that it could also be γ-Fe_2O_3 should be considered, because the two are indistinguishable by usual diffraction methods.) On all four surfaces (*A*, *B*, *C*, and *D*), Fe_3O_4 was considered present and gave an apparent maximum diffraction intensity for scuff-recovered and a decreased intensity for scuffed conditions as judged from the number of lines and from the changes in visual intensity. The identification of Fe_2O_3 on surfaces *B*, *C*, and *D* was questionable. Iron carbide was identified on the scuffed surface *D* and questionably identified on scuff-recovered surface *C*. The intensities were greater for the scuffed than the scuff-recovered surface. No Fe_3C was identified on the unscuffed surfaces. The major reaction product in the scuff debris recovered in the used oil was α-Fe_2O_3, mixed with α-Fe of small crystalline size. Some unidentified diffraction lines were also observed.

The results of the determination of crystallite sizes are presented in Fig. 4, and the X-ray patterns showing the differences are shown in Fig. 5. The hardness of the four surface conditions is also shown in Fig. 4; maximum hardness existed on the scuff-recovered surface.

Electron diffraction examination of specimens cut from an unscuffed S.A.E. 4140 steel cylinder operated more than 100 hr. with cast-iron rings and thoroughly cleaned with solvents on the outermost surface first produced the two diffuse halos characteristic of the Beilby layer and the diffraction pattern of graphite. Light abrasion with 00 emery, in successive steps under benzene, exposed metal that gave the diffraction patterns of α-Fe, Fe_3O_4, and Fe_2O_3. Oxides were thus detectable to depths estimated near 0.001 in. Oxidation during the abrasion was proved negligible by the same treatment of control specimens.

This oxidation is shown in more detail in Fig. 6 and is labeled depth of oxygen penetration. Oxides existed beneath the surface of the metal everywhere the cylinder had been rubbed. The top $\frac{1}{8}$ in. of the barrel was subjected to conditions within the combustion chamber but to no rubbing during engine operation because it was above the region of piston contact. In that top area the oxide existed only as a tenacious blue surface film under a carbon deposit. Variation in surface hardness is plotted on the same abscissa as the oxygen data in Fig. 6. These data were taken at five representative points along the barrel length.

Chromium Surfaces:

The chromium-plated cast-iron rider that was reciprocated against the steel slider contained only three distinguishable surface conditions at the end of the run, namely, worn unscuffed, burnished, and normal scuff. Unrun-surface analyses were obtained from another rider.

The chromium pattern predominates in all the surface X-ray diffraction patterns. Diffraction examination showed no presence of any chromic oxide, Cr_2O_3, on the unscuffed surface, although this surface was expected to contain at least a small amount of this oxide, possibly in the amorphous condition. The crystalline compounds Cr_2O_3 and chromium carbide, Cr_3C_2, were easily identified on the burnished and scuffed surfaces. A number of diffraction lines that stand alone and belong to none of the previously mentioned compounds may be parts of the diffraction patterns of Fe_2O_3, Fe_3O_4, the chromium nitrides, Cr_2N and CrN, the chromium carbides, $Cr_{23}C_6$, Cr_7C_3, and also Fe_3C (on the scuffed surfaces). No completely reliable quantitative trends could be found because of some difficulty in rating diffraction intensities, but the intensities of Cr_2O_3 and Cr_3C_2 appear to be greater on the scuffed than on the burnished portion. Under the microscope, patches of loose-appearing material with the color and texture of red and black iron oxides and giving dissolution reactions for iron were observed in the surface depressions, apparently as left after pickup from the mating steel slider.

The variations in hardness among the different surface conditions are graphically illustrated in Fig. 7. Crystallite-size determination showed no large differences. A decrease in crystallite size was expected approaching scuff, and such a trend was indicated, but the differences were within experimental error. The sizes were of the order of 14×10^{-7} cm.

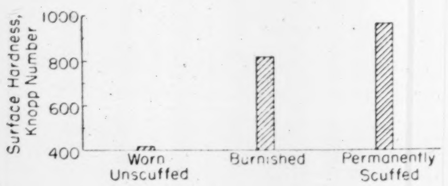


FIG. 7.—Relation Between Hardness and Surface Condition of Chromium-Plated Cast Iron Rider Specimen.

Electron-diffraction examinations of unscuffed chromium-plated rings taken from steel aircraft-engine cylinders produced no usable patterns. Only high background was observed. Under the microscope, the surfaces had the appearance of having a high polish. X-ray diffraction of these surfaces also showed considerable diffuse scatter together with very diffuse and faint lines whose approximate spacings were those of Cr_2O_3. Electrographic analyses identified chromium and iron, and a series of reactions by metallographic reagents strongly indicated the presence of the compound $Cr_2O_3 \cdot FeO$.

Cast-Iron Surfaces:

Two cast-iron slider specimens were examined after removal of the graphite coating over the underlying solid surface that had been subjected respectively to a normal and to a prolonged run-in procedure. The prolonged run-in increased load-carrying capacity. An X-ray diffraction pattern from an unrun surface showed the presence of α-Fe and one weak ring (interplanar spacing, 4.5 Å) that coincides with the strongest line of the graphite pattern. A number of weak lines and one strong line coincide with all the strong lines of Fe_3C, and several unidentified lines also show. On all the surfaces, α-Fe was observed. The presence of graphite was more completely indicated on the worn and scuffed surfaces.

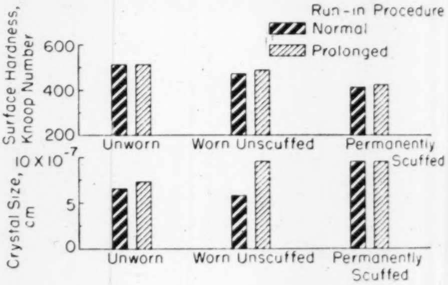


FIG. 8.—Relation Between Surface Changes And Surface Condition Of Cast Iron Slider Specimens.

Hardness values showed decrease as scuff condition was approached (Fig. 8) and crystallite sizes increased.

No examinations were made of used unplated cast iron piston rings; the information presented by Norwick and Brockway (**15** and **16**) is considered sufficient for comparison. These references discuss the detection of the presence of graphite and oxides on surfaces that were in sliding contact during engine operation.

Correlation of Results:

In general, the results may be divided into three classes: chemical reaction, material hardening, and alteration of crystallite size. The chemical and physical changes identified on surfaces from the reciprocating slider were superficial compared with the changes wrought on surfaces during engine tests. The depths of the changes were as follows:

Specimen	Depth of Change, in.
Reciprocating slider	0.00001 to 0.0001
Aircraft-engine-cylinder segment[a]	0.0001 to 0.001

[a] Except that 0.0001 in. may be maximum for chromium plate.

On all specimens examined, a certain amount of metallic smear and crystal bending was observed on or just under the surface.

The compounds present on engine-cylinder-barrel surfaces were present also on the slider surfaces of the same material. The only differences in the findings were that the various chemical compounds and phases were more easily identified on the slider specimens, and the cylinder-barrel surfaces definitely showed carbon and the Beilby layer. The diffraction patterns from the surface material on engine specimens often tended to be diffuse.

The strength of the Fe_3O_4 diffraction pattern from the scuff-recovered steel and the strong evidence of the Fe_3C from the scuffed steel indicated that Fe_3O_4 can be associated with scuff resistance and that Fe_3C can be associated with scuff. Caution should be used, however, in basing such a comparison on the evidence in this investigation alone. Evidence for the association of different oxides with different surface conditions may be found in (22 and 18). A specially prepared Fe_3O_4 surface was found to have excellent properties, partly, at least, because it allowed rapid surface-profile break-in without subsequent high wear or scuffing. This oxide was desirable for an extremely fine and granular structure firmly rooted to the base material. It was not too tenacious to resist abrasion, and broke down into a very fine polishing medium during rubbing (22). Whenever in the preparation of the oxide surface the red Fe_2O_3 formed, unfavorable results were obtained. The red oxide was soft and non-adherent (18). The fact that these two oxides of iron have very different wearing properties has further been established at the National Advisory Committee for Aeronautics subsequent to this research by measurements of the coefficient of kinetic friction (13). The coefficient of Fe_2O_3 was reported to be approximately that of dry steel and independent of load, while properly prepared Fe_3O_4 had a low coefficient depending upon the load.

The reason for this difference in surface-rubbing characteristics of these two oxides is not exactly known. Either oxide may form on a given surface depending upon temperature and pressure relations. The number of Fe_3O_4 nuclei developed in unit volume in unit time is relatively large and the velocity of crystallization is small, and this oxide may occur as an impalpable powder. The red Fe_2O_3, however, is always crystalline (14, pp. 740, 741, 776).

The engine specimens represented run-in and wear conditions entirely different from those imposed on slider surfaces. Mixed hot gases, flame, impact loading of portions of the cylinder surface, and high sliding speeds were considerably different from the room atmosphere, simple frictional heat, slow speed, and steady loading of the slider. Yet both the engine and slider surfaces exhibited the same kinds of change. The long period of operation of the engine, however, affected the depth to which oxygen penetrated.

The occurrence of oxidation in depth only in regions of surface rubbing suggests the occurrence of minute or microscopic voids as faults (or cracks) within the atomic structure or as intergranular voids (2, 6, 11, 24).

The density of an uncast metal during working decreases (**10**, **17**), possibly as a result of the propagation of structural faults or the formation of internal voids. Such a decrease should favor penetration and diffusion of other elements, particularly gaseous ones, into the metal. Such relatively deep oxygen penetration is less in agreement with the usual concentration formula, which limits the thickness of an oxide film, than with an activity-gradient formula (**7**).

The wear profile of engine cylinders showed a depression, or highest wear, at the top of compression-ring travel. In this region of high wear where less oxygen penetration seemed to have occurred, the surface had been worn away almost as fast as the penetration progressed. Farther down the length of the cylinder barrel, the rate of oxygen penetration seemed to exceed the rate of wear. As a result, oxides formed within a relatively pliable matrix of steel and possibly gave rise to a desirable surface, combining pliability with decreased abrasion, which was continually renewed under the slow abrasion of operating wear.

Breakdown of lubricant under excessive conditions may account for an amount of carbon sufficient for formation of the carbides. The formation of a carbide-rich phase and the increased surface hardness on boundary-lubricated steel against cast iron is indicated in (**20**). The presence of unidentified diffraction lines and a disappearance of the Fe_3C structure in the analysis of the scuff debris compared with the scuffed slider surface may mean the presence of the carbon in the former as a differently compounded molecular structure.

The crystallite size and the hardness of the wearing surfaces are possibly as important as the chemical reactions. The relations between crystallite size and hardness for steel, chromium, and cast iron vary as shown in Figs. 4 and 8 and are somewhat complex. The average hardness of the S.A.E. 4140 steel for the scuffed and scuff-recovered surfaces was greater than the average hardness of the unscuffed surfaces, and by similar comparison the crystallite size was smaller. In conjunction with these facts, the formation of the iron oxides and carbide on the scuffed surfaces is noted. If the change in hardness is to be attributed to one of the other changes, it seems to follow chemical reaction best. The occurrence of Fe_3O_4 in its greatest quantity coincided with the greatest hardness on the scuff-recovered surface, and a drop in hardness on the scuffed surface coincided with an indicated reduction in the amount of Fe_3O_4. This concept of a general relation of hardness to chemical reaction may or may not be weakened, however, by the occurrence of Fe_3C with the lower hardness of the permanently scuffed surface, depending upon the extent of the removal of possibly embrittled oxide-carbon-iron layer by the scuffing.

The hardness of the chromium surface increased markedly for the burnished surface condition and corresponded with the formation of crystalline Cr_2O_3 and Cr_3C_2. The cast iron softened throughout the runs and the crystallite size increased. The end result at scuff was approximately the same for normal and for prolonged run-in. The prolonged-run-in surface, having the higher load at failure, showed slightly greater softening than the normal-run-in surface. The little change in crystallite size for the worn-unscuffed surface under prolonged run-in seems more compatible with increased resistance to failure. Chemical changes may account for the reversed trend for this material with respect to the over-all trend associated with the other materials, although such changes were undetectable in this case. The graphite present on the cast iron may wear differently, for example, after reaction with moisture, or any of the interstitial atoms, hydrogen, nitrogen, carbon, and with oxygen under various conditions. Relatively, an opposite trend is indicated for any one of these materials when the change in hardness is compared with change in crystallite size between unscuffed and scuffed surface conditions.

SUMMARY OF RESULTS

The following observations were made as a result of examinations of metallic surfaces that had undergone rubbing under conditions of partly boundary and partly hydrodynamic lubrication. Changes found on steel, chromium, and cast iron varied with the run-in and scuffed surfaces. The principal chemical reactions were oxidation and carburization with the following reaction products:

Specimen	Surface Condition	Material Identified on Surface	Questionable Identification[a]
S.A.E. 4140 steel reciprocating slider	Unrun	α-Fe	Fe_3O_4
	Worn unscuffed	α-Fe	Fe_3O_4, Fe_2O_3
	Scuff recovered	α-Fe, Fe_3O_4	Fe_3C, Fe_2O_3
	Scuffed	α-Fe, Fe_3C, Fe_3O_4	Fe_2O_3
S.A.E. 4140 steel aircraft-engine cylinder barrel	Worn unscuffed		
	(Superficial face)	C (graphite), Beilby layer	Mixed oxides
	(Less than 0.001 in. below surface face)	α-Fe, Fe_3O_4, Fe_2O_3	
Chromium-plated cast-iron rider	Worn unscuffed	α-Cr	
	Burnished	α-Cr, Cr_2O_3, Cr_3C_2	
	Scuffed	α-Cr, Cr_2O_3, Cr_3C_2	
Cast-iron reciprocating slider	Unrun	α-Fe	Fe_3C
	Worn	α-Fe	C (graphite)
	Scuffed	α-Fe	C (graphite)

[a] Lines best fit pattern of material listed.

Hardness of materials examined varied with degree of surface condition. The crystallite sizes were in the order of 10^{-7} cm. The sizes changed with condition of operation.

Some relations are established between the formation of (*a*) these surface films and reaction products, hardness and crystallite size, and (*b*) the operating surface conditions on the materials investigated, as follows:

1. For steel the lower oxide, Fe_3O_4, with a higher hardness coexisted with a run-in condition, while the higher oxide Fe_2O_3 and iron carbide coexisted with surface breakdown. Oxygen penetration and higher hardness accompanied a long-time well run-in condition to a greater depth than with short-time run-in.

2. For chromium-plate, crystalline oxide Cr_2O_5, carbide Cr_3C_2, and greatly increased hardness coexisted with surface burning or breakdown.

3. For cast iron, some softening with the formation of surface graphite coexisted with both higher load-carrying capacity and with surface breakdown. For the prolonged run-in procedure, with higher load-carrying capacity, crystalline growth occurred only with scuffing.

REFERENCES

(1) Charles Sanborn Barrett, "Structure of Metals," McGraw-Hill Book Co., Inc., New York, N. Y., pp. 142–143 (1943).

(2) W. L. Bragg, "The Structure of a Cold-Worked Metal," *Proceedings*, Physical Society (London), Vol. 52, Part I, No. 289, January 1940, pp. 105–109.

(3) F. P. Bowden and T. P. Hughes, "The Friction of Clean Metals and the Influence of Absorbed Gases. The Temperature Coefficient of Friction." *Proceedings*, Royal Society, (London) Ser. A, Vol. 172, No. 949, August 3, 1939, pp. 263–279.

(4) F. P. Bowden, A. J. W. Moore, and D. Tabor, "The Ploughing and Adhesion of Sliding Metals," *Journal of Applied Physics*, Vol. 14, No. 2, February, 1943, pp. 80–91.

(5) F. P. Bowden and D. Tabor, "The Theory of Metallic Friction and the Role of Shearing and Ploughing," *Bulletin No. 145*, Commonwealth of Australia Council for *Scientific Industrial Research*, Friction and Lubrication Rep. No. 1, Part I, Melbourne, 1942.

(6) C. Crussard, "Défauts de Structure et Propriétés Mécaniques des Métaux." Contribution à l'étude de la Structure Mosaique, *Métaux*, corrosion, usure, Vol. XX, Nos. 225–226, May–June, 1944, pp. 56–66.

(7) L. S. Darken, "Diffusion in Metals Accompanied by Phase Change," Tech. Pub. No. 1479, Am. Inst. Mining and Metalurgical Engrs., August, 1942.

(8) Kurt Dies, "Fretting Corrosion, A Chemical-Mechanical Phenomenon," Engineering Digest, Vol. 5, No. 11, November, 1944, p. 324. (From V. D. I. Zeitschr., Vol. 87, No. 29–30, July 1943, pp. 475–476.)

(9) G. T. Finch, "The Structure of Sliding Surfaces," *Engineering*, Vol. 159, No. 4131, March 16, 1945, p. 215.

(10) P. Goerens, On the Influence of Cold-Working and Annealing on Properties of Iron and Steel, Parts I and II, *Journal*, Iron and Steel Inst., Carnegie Scholarship Memoirs, Vol. 3, pp. 320–434 (1911).

(11) A. A. Griffith, "The Phenomena of Rupture and Flow in Solids," *Philosophic Transactions*, Royal Society (London), Ser. A, Vol. 221, March, 1921, pp. 163–198.

(12) T. P. Hughes and G. Whittingham, "The Influence of Surface Films on the Dry and Lubricated Sliding of Metals," *Transactions*, Faraday Soc., Vol. XXXVIII, Part I, No. 249, January, 1942, pp. 9–27.

(13) Robert L. Johnson, Douglas Godfrey, and Edmond E. Bisson, "Friction of Solid Films on Steel at High Sliding Velocities," N.A.C.A. TN No. 1578, April, 1948.

(14) J. W. Mellor, "Ferric Oxide. Ferrosic Oxide; or Magnetic Oxide of Iron," Vol. XIII of A Comprehensive Treatise on Inorganic and Theoretical Chemistry, Part 2 of Iron, Ch. LXVI, Sec. 29–30, Longmans, Green and Co., pp. 731–831 (1934).

(15) A. S. Nowick and L. O. Brockway, "An Electron-Diffraction Examination of Cast-Iron Piston Rings from Single-Cylinder Aircraft-Engine Tests," N.A.C.A. ACR No. E4J25, 1945.

(16) A. S. Nowick and L. O. Brockway, "Occurrence of Iron Oxides on Cast-Iron Engine Surfaces after Operation," N.A.C.A. ACR No. E5L18, 1945.

(17) Hugh O'Neill, "The Effect of Cold-Work upon the Density of Crystals of α-Iron," *Journal*, Iron and Steel Inst., Vol. CIX, No. 1, pp. 93–108; Discussion, pp. 109–120; Correspondence, pp. 121–128 (1924).

(18) Max M. Roensch, "Piston-Ring Coatings and Their Effect on Ring and Bore Wear," *SAE Journal*, Soc. Automotive Engrs. (*Transactions*), Vol. 46, No. 5, May, 1940, pp. 221–228.

(19) Samuel J. Rosenberg and Louis Jordon, "The Influence of Oxide Films on the Wear of Steels," *Transactions*, Am. Soc. Metals, Vol. 23, September, 1935, pp. 577–598, Discussion, pp. 598–613.

(20) B. W. Sakmann, "Metallurgical Transformations in Metal Surfaces under Conditions of Boundary Lubrication," Abs. contributed paper, Am. Phys. Soc. (New York), Jan. 19–20, 1945. Phys. Rev., Vol. 67, Nos. 5–6, March 1–15, 1945, p. 200.

(21) Macy O. Teetor, "Load-Carrying-Capacity Phenomena of Bearing Surfaces," SAE *Journal*, Soc. Automotive Engrs., (*Transactions*), Vol. 47, No. 6, December, 1940, pp. 497–503.

(22) Macy O. Teetor, "The Reduction of Piston-Ring and Cylinder Wear," *SAE Journal*, Soc. Automotive Engrs., (*Transactions*), Vol. 42, No. 4, April, 1938, pp. 137–140, 156.

(23) Wells A. Webbs, "The Influence of Iron Oxide on Wear of Rubbing Surfaces," *Science*, Vol. 99, No. 2575, May 5, 1944, pp. 369–371.

(24) C. Zener and J. H. Holloman, "Plastic Flow and Rupture of Metals," *Transactions*, Am. Soc. Metals, Vol. XXXIII, pp. 163–215 (1934).

DISCUSSION

Mr. John M. Lessells.[1]—Those of us who are interested in fatigue believe we do not know too much about the subject. As regards wear, possibly less is known.

It would be interesting to learn if the authors have tried to correlate their results with the more fundamental work of Bowden[2] and his associates. Bowden, as you recall, has explained why different results are obtained from similar and dissimilar metals in contact and has advanced certain theories regarding such.

Bowden[3] also shows that with metals in contact very high temperatures are obtained, so wear could really be considered a problem of local melting. In view of this it would be of interest to know how the authors measured temperatures.

As an engineer I am, of course, interested in the order of merit of the different surfaces. If we take the particular case of a cylinder liner and the piston ring there are as many ideas as regards suitable materials as there are engine builders. Does the author see any advantage in the use of a porous chromium ring in contact with a cast iron?

Mr. J. W. Good (*author*).—A consideration of fatigue together with wear, in connection with this paper, would lead through a large number of points of discussion covering fatigue, metallurgy, and fundamental structural properties of metals as a whole. Of course, there is fatigue here, in a way. The loading is attended by very rapid application and release over finite points of contact, whether there is sliding or rolling contact, or just sliding contact under load and wear. So fatigue is a phase of this problem which has to be considered very seriously.

We have done some work which includes correlation of our findings at the NACA with Mr. Bowden's work and that of others in connection with a recent program in the study of coefficients of kinetic friction and wear. This work, under some other authors, is already published in part and is continuing.[4]

It can be found in the details of our paper that a thin amorphous or structureless layer was found on the outermost face of the specimens, for example, the aircraft engine cylinder panel. This layer, of metal and whatever else may have been absorbed in its formation, is structureless, and is most probably resultant from the local melting encountered at the points of sliding contact at the surface.

It was known before the program was started that these high temperatures are very rapidly diffused through the surface as the heat penetrates into underlying metal. It was felt, also, that a condition of layer-like surface strata could and should exist at and below the surface. These strata would represent phases formed at different temperature regions and by different aggregate, physicochemical reactions. The importance attached to these strata, which must carry the load by supporting the melted-through surface atoms, led us to measure the temperature of the specimen mass immediately underlying the sliding surfaces, particularly with reference to mass-temperature *versus* surface load effects. Slide-wire contacts were used in this case, with ordinary thermocouple junctions.

---

[1] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[2] F. P. Bowden, L. Leben, and D. Tabor, "The Sliding of Metals, Frictional Fluctuations, and Vibrations of Moving Parts," *Engineer*, August 29, 1939, p. 214.

[3] F. P. Bowden and K. E. Ridler, "A Note on Surface Temperature of Sliding Metals," *Proceedings*, Cambridge Philosophical Society, p. 431 (1935).

[4] Robert L. Johnson, Douglas Godfrey, and Edmond E. Bisson, "Friction of Solid Films on Steel at High Sliding Velocities," Nat. Advisory Committee for Aeronautics, TN No. 1578.

In answering the question about the comparison between the chromium-plate and cast iron, I can say that dense chromium-plate was used. Whether the one is better than the other I do not think I would want to say from these data, although there does not seem to be much difference.

MR. J. T. MACKENZIE.[5]—I should like to know whether the author has the composition and microstructure of the particular cast iron used?

MR. GOOD.—I can only say, concerning the composition and microstructure of the particular cast iron used, that it was that of standard liners used in military aircraft during a period of quantity production.

MR. P. A. BECK.[6]—This very interesting paper seems to establish the fact quite clearly that oxidation does take place at the contact area of two rubbing surfaces. The phenomenon appears to be quite general. Years ago, in running fatigue tests on lead alloys, we noticed that the surface of the specimen end, where it was clamped into the holder and undoubtedly rubbing against the holder during the test, gradually became covered with a reddish layer, probably lead oxide. Another phenomenon of more practical interest, may be also related to the acceleration of oxidation by rubbing. It was observed that failure in copper lead bearings in internal combustion engines is usually connected with the preferential removal of lead from the bearing alloy. The oxidation or corrosion of the lead at the bearing surface apparently takes place much more readily when the shaft is rubbing against the bearing than it does in the same oil at the same oil temperature, without actual rubbing.

The question is then why oxidation or corrosion takes place so much more readily when two surfaces are rubbing against each other. One explanation that appears plausible is that the chemical reaction rate is increased by a highly localized temperature increase which may develop in a very thin surface layer. I should like very much to have the authors' reaction to this possible theory.

MR. GOOD.—In the examples given of rubbing oxidation of lead alloys, it is noted that they all are of a chafe-type rubbing. That is, particles of metallic solid become abraded and oxidized at the same time. The action in these cases is accompanied by the removal of solid material from the surface during the action. Temperature activation will naturally accelerate the chemical reaction, in most cases, in general. The point that must be made, however, is that the chemical reactions brought out in this paper take place in a manner of permeation and phase change rather than of corrosion.

MR. O. W. ELLIS.[7]—Some years ago we presented a paper on wear tests on ferrous metals.[8] The results of these tests are not very closely connected with the work that has been discussed in the present paper; nevertheless there were one or two that may be of interest.

Our work on wear was carried out in ball mills in which water was present.

---

[5] Technical Director, American Cast Iron Pipe Co., Birmingham, Ala.

[6] Associate Professor of Metallurgy, Department of Metallurgy, University of Notre Dame, Notre Dame, Ind.

[7] Director, Dept. of Engineering and Metallurgy, Ontario Research Foundation, Toronto, Ontario, Canada.

[8] *Transactions*, Am. Soc. Metals, Vol. 30, p. 249 (1942).

In one series of experiments we introduced a number of forged steel balls into a mill, hermetically sealed, which was filled with nitrogen before the test was started. It so happened that, by accident, one of the two steel diaphragms that closed the mill had a hole in it about two inches in diameter, so that to seal the mill on the side where the hole was present we placed a heavy solid rubber diaphragm about $\frac{1}{2}$ in. thick.

When the mill was operated the temperature of the charge rose. On this account the pressure in the mill increased and, before we knew it, some of the contents of the mill were forced out of the mill and thrown around the laboratory.

On the other hand, when we ran the mill with air or oxygen in it, despite the fact that the temperature rose to the same extent, reaction between the steel balls and the oxygen in the mill resulted in a lowering of the pressure within the mill, so that the rubber diaphragm bulged in towards the inside of the mill.

In another series of tests, we used a small porcelain jar as our mill. In this case we took 10 steel balls, two of each of five alloys, varying in carbon content from 0.50 to 2.50 per cent. We ran these alloys first in an atmosphere of nitrogen, and then in an atmosphere of oxygen; the wear increased with the carbon content. When tests were carried out with balls of similar carbon content, but with 15 per cent chromium present in all the alloys, the results were rather astonishing. In oxygen, the high-carbon chromium alloy wore relatively rapidly, but the low-carbon chromium alloys wore relatively slowly. In nitrogen the loss of weight was about the same in all the alloys, but—and this is the point I want to make perfectly clear—in nitrogen this loss of weight of the chromium steels was of the same order as that of the straight iron-carbon alloys. In other words, in nitrogen the straight iron-carbon alloys behaved as though they contained chromium, and in oxygen the low-carbon high-chromium alloys behaved in a fashion similar to what they did in the nitrogen.

MR. GOOD.—The results from the ball mill, using nitrogen and oxygen, are very interesting. A little might be said here about some of the work that was done at the National Advisory Committee for Aeronautics with a nitrided steel piston ring. The surface materials are very hard with apparently very low wear. Under high temperatures and vacuum, the nitrogen completely left the surface. The tests later, on nitrided rings from engines, gave similar results. Over a long period of operation under high temperatures, the nitrogen apparently migrated to the surface. The initial optimum surface condition was thereby removed. The results with oxygen in your ball mill tie in very nicely with the conditions of oxidation we found under run-in and wear.

SYMPOSIUM ON USEFULNESS AND LIMITATIONS OF SAMPLES

INTRODUCTION

By H. F. Dodge[1]

This Symposium on the Usefulness and Limitations of Samples is the first to be presented by Committee E-11 on Quality Control of Materials.

This Committee came into being just two years ago largely as a result of increased emphasis on and expanded use of quality control techniques during the war, taking over and enlarging upon the duties of Subcommittee IX, of Committee E-1 on Interpretation and Presentation of Data. It was organized to promote the knowledge of quality control methods and their application to A.S.T.M. problems. Here, by quality control methods is meant those methods that have been developed on a statistical basis to control the quality of product through the proper relation of specification, production and inspection as parts of a continuing set of operations.

During the past year the Committee has been urged to hold a general session on some of the subjects that have been under consideration. Furthermore, the Committee was informed that what was wanted was a discussion of topics at the engineer's level; not statistical discussions of Graeco-Latin squares or tests of alternate hypotheses. Accordingly, the task of organizing the session was assigned to two men well known in A.S.T.M. for their practical-mindedness and basic understanding of A.S.T.M. problems, Mr. Arthur W. Carpenter and Mr. A. G. Ashcroft. Both have had a close interest in applications of statistical methods.

As a result of their efforts we have on our program three real experts, experts not merely in the techniques of quality control but more important, I think, experts in the art of getting ideas across to others.

All of us, when dealing with materials, are continually up against the problem of sampling and of making use of results obtained from samples of one kind or another. At any one time we may be faced with one of two general types of problems. First, what is the quality of this specific batch of material that is before us now, how closely may we determine its properties, and how shall we interpret the test results in making a decision regarding its disposition? Second, how is the manufacturing process, which is turning out this material, behaving, is it essentially stable or is it subject to erratic variations, can something and should something be done to the process to provide greater uniformity?

We try to solve both types of problems by taking data, but many questions arise which must be answered, both in the collection of and in the analysis of data. How do we go about sampling bulk material as distinguished from a product made up of individual pieces or articles? If the material sampled is not homogeneous (and it usually is not) how should we spread our sample over the whole? Having obtained a given set of data, how reliable are our results? How large an error may have been introduced because of the size of our sample and because of the method of sampling?

---

[1] Chairman of Committee E-11 on Quality Control of Materials; Quality Results Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

Then again, we know that our test data are subject to two influences: first the variations in the material itself, and second, the variations due to the testing operation. The question often arises: How much of the total variation is attributable to material and how much to testing?

Further, as we make tests on successive consignments of material from different sources, we often have the question: How much testing is necessary so that good material will generally be accepted and poor material rejected? If material from any one source is consistently good, are there any general principles governing the degree to which we can reduce the amount of testing as compared with that required when the quality is erratic?

These are some of the questions with which we are continually confronted and there are certain general principles that may be used as guides in determining the character and quantity of numerical data that should be obtained under a given set of conditions.

The three papers in our program consider some of these broad questions and indicate ways in which the methods of statistics can be of aid to us as engineers in answering them. Probability laws are invoked, but consideration is given to the use of empirical criteria that are suited to the world of experience.

SAMPLING AND ITS UNCERTAINTIES*

By S. S. Wilks[1]

Synopsis

There are two stages in the sampling of materials. Stage I deals with the problem of variability of test results due to selecting only a small fraction of a lot to test. Stage II deals with the problem of variability of test results due to making only a small number of measurements on the material selected at Stage I. Stage II is the more fundamental because at this stage the validity of whatever measurement operation is to be used in testing must be established. Stage I sampling makes use of measurements validated at the Stage II level.

In the present paper mathematical and statistical methods are discussed for analyzing sampling results and describing sampling laws at each stage of sampling. Precision and accuracy of measurements are discussed in connection with Stage II sampling, and statistical devices for testing whether conditions exist under which they can be measured are described briefly. Two types of Stage I sampling are considered: (*a*) sampling from a lot of product pieces, and (*b*) sampling from a bulk of material. In each case sampling laws under random sampling conditions are described. In particular, the sampling law for number of defectives occurring in a sample from a lot of product pieces containing a specified number of defectives is given, with comments on its application to inspection sampling. In sampling from lots of bulk material the sampling laws for the least and greatest measurements in a sample and their application in making inferences about the lot are described. The sampling law of sample averages and its use in making inferences about the lot is treated.

Representative sampling as a method of overcoming nonuniformity in lots and the conservative nature of inferences from representative samples are considered. Comments are also made about composite sampling and the type of information destroyed when it is used. Some attention is given to general sources of bias in sampling.

No attempt has been made to go into great detail in this paper. The purpose has been rather to discuss principles with enough references to literature to enable the reader interested in technical details and applications to find what he needs.

The idea of selecting samples from supplies or lots of products or materials for the purpose of obtaining information about characteristics of these aggregates is a very old one indeed. We all know that when there is nonuniformity or variability in the supply, the information which a sample of the material furnishes about the supply may have a considerable amount of uncertainty about it. Such a sample may consist only of some of the "best" of the supply or some of the "worst," depending on who selected the sample, how he selected it, and for what purpose. Every housewife who has purchased a box of strawberries with neatly arranged rows of large luscious berries on top knows what a selected sample of some of the "best" of a supply often means. Sampling which may do no better job of accurately describing a supply or lot than this is innocently practiced in many fields by persons who may be either unaware of the capriciousness of sampling or careless about guarding against it. Selective sampling of this kind will probably be practiced by human beings as long as it has any persuasive effect on the actions of others, or as long as they do not fully realize the hazards in drawing conclusions from such samples.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Professor of Mathematical Statistics, Princeton University, Princeton, N.J.

But in spite of such practices as these there is a growing awareness on the part of many who use sampling methods that sampling plays a very fundamental rôle in investigation, testing, and experimentation. Examples of such persons are quality control engineers, government census experts, agricultural scientists, public opinion pollers, market research men, crop estimators, wool samplers, and highway traffic analysts. They are coming to realize more and more clearly that correct conclusions regarding characteristics of a supply or lot of products or materials depend in a very basic manner on two aspects of an over-all procedure: (I) the method by which the sample of materials or products is selected, and (II) the process by which information is obtained or measurements are made on the sample of materials, once they are selected. These may be regarded as two equally important *stages* of an over-all sampling process: Stage I deals with the problem of variability of test results due to selecting only a small fraction of the lot to test, and Stage II deals with the problem of variability of test results due to making only a small number (often only one) of measurements on the material which is selected at Stage I. Variability in the final results of the test can and does enter at each of these two stages. Stage II is the more basic of the two stages in the sense that it is at that stage when the degree of *accuracy* and *precision* (sometimes called *validity* and *reproducibility*) of the actual measurements themselves must be established. These cannot be established by merely assuming that the measurement obtained in a single test represents what we are usually tempted to think it represents. If measurements are repeated, variability is found and usually some of this variability is attributable to *assignable* causes (that is, causes which can be located) which may not have been suspected initially. Stage I sampling laws depend on making use of measurements whose accuracy and precision has been established at the Stage II level.

As a result of this growing sampling consciousness, more effort is now being made to establish Stage I and Stage II sampling procedures on a scientific basis, so that when sampling is carried out in an objective manner in accordance with a specified procedure the reliability of conclusions from the sample regarding characteristics of the lot will be known. In other words the attitude in many quarters is that if we have to live with the vagaries of sampling then we must learn something about the conditions under which these vagaries can be evaluated and then abide by these conditions as closely as we can.

Some of the progress which has been made toward more scientific sampling at Stages I and II is experimental. Investigators have found by repetitions of their sampling how much their results vary from sample to sample. On the other hand, a substantial part of the progress is mathematical. Theoretical sampling laws have been worked out mathematically and many of them have been checked experimentally. Mathematical methods furnish a simple and cheap way of working out these laws, particularly in the simpler cases. Sampling laws are founded on the theory of probability—a discipline which had its beginning 300 years ago in theoretical studies of the possible outcomes of games of chance. This discipline has been gradually developed into a mature branch of mathematics which now serves not only as the basic mathematical tool for sampling theory but also for quantum mechanics, genetics, and various other branches of science.

The present paper deals primarily with some mathematical and statistical aspects of sampling at both Stages I and II and their interpretation from the point of view of the testing engineer. The experimental and immediately practical aspects of the subject are covered in other papers on this program.

Since Stage II sampling is the more basic of the two stages we shall discuss it first. It is closely associated with the experimental or testing process by which a measurement is obtained. A full discussion of it for a specific problem in any given field would involve making use of detailed technical knowledge in that field. We cannot hope to attempt such a discussion. But there are certain general principles of a mathematical statistical type which will be discussed briefly.

1. STAGE II SAMPLING—PRECISION AND ACCURACY OF MEASUREMENTS

Consider a process of making a certain physical measurement on an object repeatedly. If we were to get the same value of each measurement we would have perfect *precision* of measurement. If in addition the measurement always had a value equal to the *actual* or *true* value (assuming for the moment that there is one) of the measurement, then we would also have perfect *accuracy* of measurement. In practice, we rarely, if ever, have perfect precision or perfect accuracy. Furthermore, if we take the liberty of assuming that the object has a true value of the measurement, then we often *do not know* its value. This means that, in practice, we have to make inferences about degree of precision and degree of accuracy of measurements from the measurements themselves. Precision is the more fundamental concept and conditions under which it has meaning and can be measured have to be established before one can talk about accuracy.

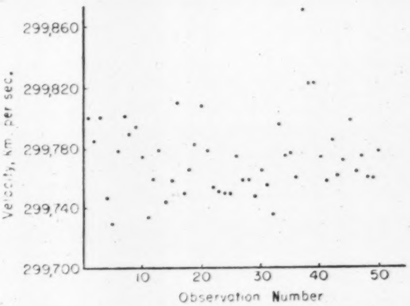


FIG. 1.—Graph of First 50 of Michelson's Light Velocity Measurements Plotted Against Order in Which Measurements Were Made.

In practice, if we take a sequence of repeated measurements on an object the measurements have some degree of uncertainty about them; they vary from one to another from various causes, some of which may be determined as *assignable* and others unknown or *random*. This variation may be shown effectively in a graphical manner by plotting the successive measurements as ordinates against the order in which the measurements were made. For instance, if we take the first 50 measurements on the velocity of light (in kilometers per second) made in February, 1931, by Michelson and his colleagues (**1**)[2] and plot them against the order in which they were made, we obtain Fig. 1. Percentagewise, the variations are extremely small in this case, but they do exist and are important in astronomical work.

Now in order to have conditions under which the degree of precision of a measurement operation can be established, these ordinates must vary up and down in a *random* manner as one progresses through the sequence of measurements, showing no tendency to gradually rise or fall or jump from one level to another, or behave in any other "non-random" manner. If there is an upward or downward trend or some other non-random characteristic of the sequence of measurements, the chances are high that certain unsuspected *assignable causes* of variations have crept in as the measurements were made to cause such trends or other peculiarities to appear. When such causes are found and eliminated the tendency is for further measurements to vary more "randomly."

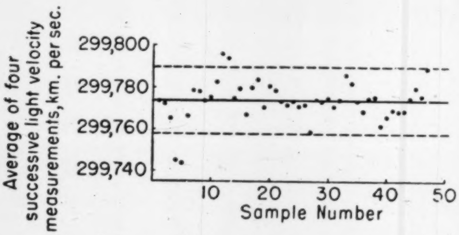


FIG. 2.—Control Chart of Averages of 47 Successive Samples of 4 of Michelson's Light Velocity Measurements.

In practice, the basic question which arises is this: Given a sequence of measurements, how can we tell beyond a reasonable doubt that the repeated measurements are behaving as though some assignable cause or factor had crept in to upset "randomness"? In many cases it will be graphically obvious that the measurements have a significant trend in them or have too many excessively large or small values. In the less obvious cases (of which Fig. 1 is an example) there are mathematical statistical devices which, when applied to the sequences of measurements, will provide such indications. The simplest and most effective is the *control chart* devised by Shewhart (**2**) which shows averages of successive sets or *samples* of 4 or 5 measurements plotted as ordinates against the order in which the successive sets of measurements are made. Figure 2 shows an example of a control chart, constructed by Shewhart (**3**) by taking 47 successive samples of 4 of the Michelson light velocity measurements. It will be noticed that there is a *central line* and two *control limits* (an *upper* and *lower* control limit) on the control chart. These are usually determined from the information provided by a sequence of at least 25 sets of measurements, although they may be determined from previous experiments or even theoretically in certain instances. The technique by which these lines are established may be found in reference (**4**). Control charts may not only be established for averages of successive sets of 4 or 5, but for ranges (largest measurement minus smallest) of successive sets of 4 or 5, and other quantities. If the measurements are *under statistical control*, that is, no assignable causes of variation are indicated, the means will jump in a "random" fashion about the central line and between the control lines, with an occasional point (less than 1 out of 100) falling outside the control limits. On the other hand, if the measurements are not under control, too many points will fall outside the control limits or trends of one kind or another will show up in the plotted sequence of averages.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 874.

Averages of small samples (4 or 5 cases) of measurements are plotted in constructing control charts rather than individual values because in practice the points jump around in about the same way under conditions of statistical control regardless of how the individual measurements behave under these conditions. In other words, under these conditions, sample averages tend to be approximately *normally* distributed regardless of how individual measurements are distributed in practical situations.

On most of the occasions in which control charts are used discrepancies from a state of statistical control are fairly obvious to the eye. In others one can make such a judgment by seeing whether too many points are outside the control lines. Looking at Fig. 2, it will be seen that five points fall on or outside the control limits. This fact alone indicates beyond a reasonable doubt the entrance of assignable causes of variation during the period in which the successive measurements on light velocity were being made. In fact, Michelson stated one assignable cause of variation and conjectured another.

In those cases where the causes for non-randomness in a sequence of measurements are too elusive to be picked up by a control chart such as that in Fig. 2, there are other mathematical techniques for detecting the presence of assignable causes of variation. One of these utilizes *runs*, that is, lengths of rising or falling sequences of points on a control chart, or lengths of sequences of points above or below some fixed horizontal line on the chart. Another device is *serial correlation* which measures the degree to which successive points on a control chart are "chained" together. Another technique which has been used to some extent in industry and promises to be very useful in detecting and measuring the effects various causes of variation in measurements, once the probable causes or factors are identified, is the *analysis of variance*. A discussion of these techniques is beyond the scope of this paper. The interested reader should consult references (**5, 6, 7**) for details on the application of these methods.

It should be repeated that the ideal conditions under which the precision of measurement can be established require a state of statistical control, that is, the absence of assignable causes of variation. When causes of variation have been detected by control charts and other devices and eliminated, one arrives at a state of statistical control. Under these conditions the precision of measurements is really measured by the amount of variability left in the measurements. The usual index for measuring this variability is the *standard deviation*, which one can calculate from the measurements.

The idea of precision of a given measurement on an object transfers immediately to the idea of precision of a given measurement on each article in a sequence of supposedly identical articles coming off a production line. In fact, if an object is destroyed or altered by the process of making a measurement, one clearly cannot make repeated measurements on the same object. Under the circumstances one would have to make measurements on a sequence of objects which are as nearly identical "as possible." Establishing a state of statistical control in this kind of a situation by means of control charts and other devices means detection and elimination of assignable causes of variation in the manufacturing process (due to raw materials, machine operations, operators, etc.). In fact these devices serve their major function in mass production by helping to establish a state of statistical control. For a full discussion of the function of control charts under such conditions the reader is referred to (**3, 4**).

Thus far we have discussed precision of measurement and have indicated in general the conditions under which it has meaning and can be measured. Precision of measurement is the degree of reproducibility of measurements under a given set of conditions. When a state of statistical control has been established, we may say that the measurements are reproducible with a degree of precision which can be expressed numerically. The more rigidly the conditions are held constant the greater the precision of measurement. The appraisal and measurement of precision are made from statistical treatment of observations along the lines discussed in the foregoing paragraphs.

Accuracy of measurement of an object is the degree to which reproducible measurements differ from the *true* value of the object. There can be two methods of making a measurement on an object, and both methods can have high degrees of precision but different degrees of accuracy. Visualizing measurements by each method plotted in control chart fashion, one method can produce measurements which fluctuate about one level of values, and the second can produce measurements which fluctuate about a different level of values. One of these levels or neither may be the true level of values for successive measurements of the object. To actually establish accuracy of a sequence of measurements whose precision has been established it is necessary to *know* the true value of the measurement. In cases where there is no way of knowing *a priori* the true value, an attempt is often made to define it from observations obtained under specified conditions. Examples are velocity of light, acceleration due to gravity, etc. The main condition under which such a definition can be made is that the successive measurements be in a state of statistical control.

2. STAGE I SAMPLING—VARIATION IN MATERIAL DRAWN FROM A LOT

Stage II sampling problems have been satisfactorily dealt with when repeated measurements are brought into a state of statistical control with known precision and accuracy. In other words, Stage II sampling establishes validity of measurements. Stage I sampling deals with fluctuations of measurements of established validity from one piece of material in an aggregate or lot to another.

There are two main kinds of Stage I sampling problems of interest to the engineer:

(*a*) Sampling from a lot of articles or product pieces,

(*b*) Sampling from a lot of material in bulk.

We shall discuss these two kinds of sampling with particular reference to laws for describing sampling fluctuations of measurements and conditions under which these laws are applicable. We shall also try to indicate what happens to these laws when some of the conditions cannot be realized or when they are violated. Throughout the discussion we shall deal with basic principles and hypothetical illustrations rather than attempt to discuss an assortment of specific cases which arise in testing work.

(*a*) *Sampling from a Lot of Articles:*

Sampling Laws for this Type of Sampling.—In many testing situations one has a lot of N articles or product pieces manufactured under "similar" conditions—similar with respect to raw materials, manufacturing operations, etc. Each article in the lot is capable of being tested, that is, measured, with respect to one or more physical, chemical, or other characteristics which are considered important. For the sake of simplicity, let us consider only one characteristic and suppose that each product piece in the entire lot, if tested, would be classified either as a *defective* or a *non-defective* with respect to this characteristic. A sample of n pieces is taken from this lot and tested with respect to this characteristic. In this simple sampling situation there is uncertainty in the number of defectives which will be found in samples if the lot contains both defectives and non-defectives. This uncertainty is absolutely inevitable and has to be accepted. So the real question is this: Can the uncertainty be measured or evaluated or controlled and under what conditions can this be done? The answer is that a perfectly definite law can be set up which will, under certain ideal conditions, tell us with what frequencies we can expect 0, 1, 2, 3, . . . , etc. defectives to occur in the sample if we could repeat the sampling and testing many times from lots with the same composition of defectives and non-defectives. The principal condition under which such a law operates is that all pieces in the lot must have "equal chances" of being included in the sample. The law is a mathematical one and is worked out by considering every possible sample of n pieces which could be formed from the lot of N pieces. By using certain combinatorial methods, the samples containing 0 defectives are counted, those containing 1 are counted, and so on. These frequencies, each divided by the total number of samples of n pieces which can be formed from the lot of N pieces, constitute the law of uncertainty with which any specified number of defectives will turn up in the sample. This law of uncertainty is a *probability law*. It is a probability law for a characteristic of the sample which is measured by *counting*. It states the probability that 0, 1, 2, 3, or any specified number of defectives will appear in the sample. The probability law may also be expressed in *cumulative* form which would give the probability of not more than 0, 1, 2, 3, or any specified number of defectives. Such a law will, in general, depend on three quantities: N, the lot size, p, the fraction of defectives in the lot; n, the sample size.

As a simple illustrative example, Fig. 3 shows the cumulative sampling law for samples of 10 from a lot of 100 pieces, for $p = 0.1$, 0.2, 0.3, 0.4, and 0.5. Note that there is a curve for each value of p; for any given value of r (the number of defectives in the sample) the ordinate for that curve gives the probability that the number of defective pieces in the sample will not exceed that value of r. The only points on a curve for a given value of p which actually correspond to reality are those for which $r = 0$, 1, 2, . . . , 10, since it is obviously impossible to have a fractional number of defectives in a sample. As an example, it will be seen that for $p = 0.3$ (that is, for 30 defectives in the lot of 100 pieces) and $r = 4$, the ordinate is 0.86. This means that the probability is 0.86 that the number of defectives in a sample of

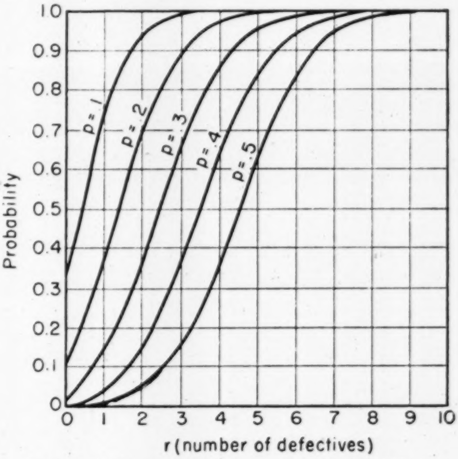


FIG. 3.—Curves Showing the Probability of Getting Not More Than r Defectives in a Sample of 10 from a Lot of 100 Having Fraction of Defectives $p = 0.1$, 0.2, 0.3, 0.4, and 0.5.

10 pieces from a lot of 100 containing 30 defectives will not exceed 4.

By varying N, p, and n one can find the sampling law of defectives for any size of lot, any sample size and any fraction of defectives in the lot. In actual practice, simplifying approximations are used to calculate the probabilities required.

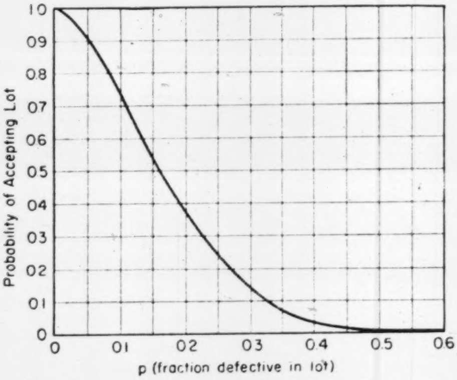


FIG. 4.—Operating Characteristic Curve for Following Illustrative Single Sampling Plan:

(*a*) Draw sample of 10 from lot of 100.
(*b*) Reject lot if sample contains more than one defective.
(*c*) Accept lot otherwise.

Inspection Sampling Plans.—These sampling laws of which Fig. 3 is a simple example constitute the foundation upon which modern *inspection sampling plans* are built. We do not propose to do more tham mention briefly here the idea of inspection sampling and that it is built on sampling laws of the type already discussed. For details of such plans the reader is referred to references (**8, 9, 10**).

There are several types of sampling plans. The simplest type is the *single sampling plan*, which specifies that a sample of a given size is to be drawn from a lot of a given size and that if the number of defectives in the sample exceeds a *maximum allowable defective number* then the lot is rejected. Otherwise the lot is accepted.

As a simple *single sampling* plan consider the following example: A lot of 100 articles is submitted for inspection. A sample of 10 is drawn at random from the lot and inspected. If the sample contains more than one defective the lot is rejected, otherwise the lot is accepted. A very effective device for showing how such a plan would work for lots with varying fractions of defectives is the *operating characteristic curve.* Figure 4 shows the operating characteristic curve for the plan described above. The data for the curve in Fig. 4 is obtained from that given in Fig. 3. The ordinate of the curve for any fraction defective in the lot gives the probability that the sampling plan would accept a lot with that particular fraction defective. For instance, the ordinate for fraction defective in the lot equal to 0.2 is 0.36, which means that the probability is about 0.36 that a lot of 100 pieces with 20 per cent of its items defective would be accepted by the sampling plan.

A slightly more elaborate type of plan is the *double sampling plan.* In such a plan provision is made for drawing an additional sample by setting up a "doubtful" range for number of defectives in the first sample, such that when the number of defectives in the first sample falls in this "doubtful" range a second sample is drawn. Thus, in such a plan provision is made to accept or reject the lot or remain in doubt on the basis of the first sample, and to accept or reject on the basis of the second sample if a second sample has to be drawn. A more complicated type of plan is a *multiple or sequential sampling plan* in which provision is made to draw two or more additional samples by setting up "doubtful" ranges of numbers of defectives for the earlier samples.

Importance of Randomness in Sampling Laws.—It is to be emphasized that the main condition for the validity of the sampling laws discussed above is that the various pieces in the lot must have "equal chances" of being included in the sample. From an operational point of view this can be achieved only by some randomizing procedure which is *equivalent to*: (*a*) assigning numbers to the pieces in the lot and corresponding numbers to chips or tags, (*b*) stirring the chips thoroughly in a bowl, let us say, and drawing a sample of chips, and (*c*) selecting the pieces in the lot which bear the numbers on the sample of chips. It is not always safe to assume that a given method of stirring and drawing chips from a bowl will produce a random sequence. To test whether it will, it would be sufficient to set up a control chart for the number of "defectives" in successive samples of a given number of chips, the chips being returned to the bowl before drawing the next sample. When a sample is drawn under these conditions we say that the sample is *drawn at random*. In practice it is quite often very difficult or indeed impossible to carry out true random sampling because some pieces in the lot cannot be reached without prohibitive effort. The usual result is that the procedure has to be relaxed by permitting some exercise of human choice. In this case the sampling then becomes subject in some degree to the weaknesses inherent in human selection. It would be difficult to measure the effect of this compromise in any given field of sampling without a great deal of experimental work based on repeated samplings. But in practice any expert sampler who knows sampling laws and the material with which he is working will be alert to all kinds of possibilities of maximizing randomization by choice of time for sampling, by employing techniques for reducing the effect of human choice in drawing the sample, and so on.

(*b*) *Sampling from a Bulk of Material:*

The Idea of a Population of Measurements.—The second main type of sampling of interest to the engineer is that in which one or more small batches are taken from a bulk of material for analysis or testing. Examples of situations in which such sampling is done are sampling of wool, coal, cement, textiles, and explosives.

In sampling problems of this type, the testing engineer is usually interested in measuring some characteristic of each small batch of material by *reading a scale* of some kind. He obtains weights, sizes, breaking strengths, crushing strengths, per cent ash in coal, per cent iron in an alloy, and so on. Again, it is assumed that the validity of the operation of making measurements has been established by consideration of Stage II sampling problems.

We shall discuss the sampling laws of such measurements as these under certain ideal conditions in which the bulk is considered as being made up of a large number of *batches* or *sample units* of equal size or volume and that in drawing a sample unit from the bulk, all sample units are "equally likely" to be drawn.

It should be pointed out here that if one considers a large lot of product pieces (as discussed in section 2(*a*)) and is interested in a test on each piece which would result in a measurement read from a scale rather than result in a defective or non-defective then we would have a situation very similar to the problem of sampling from a bulk of material as we have defined such sampling. As a matter of fact, this kind of sampling is usually called *sampling by variables* as contrasted to *sampling by attributes* for the case in which articles are classified as either defective or non-defective.

To take a typical example and elaborate further, let us consider breaking strength of yarn of a certain kind and size. A piece of yarn is taken from a bobbin and tested for breaking strength. If this operation is repeated on another piece of yarn from the same bobbin, another value would be obtained. If one imagined breaking strengths obtained for the thousands of pieces of equal length into which the bobbin of yarn can be cut, one would obtain a *distribution* of breaking strengths. We may conveniently refer to this collection of measurements as a *population* of breaking strengths. The distribution for such a *population* may be represented graphically by some kind of a curve as shown in Fig. 5. This curve is such that the area under it between any two specified breaking strengths represents the fraction of pieces of yarn having breaking strengths between those two specified values. For example, the shaded area in Fig. 5 represents the fraction of pieces with breaking strengths between *a* and *b*.

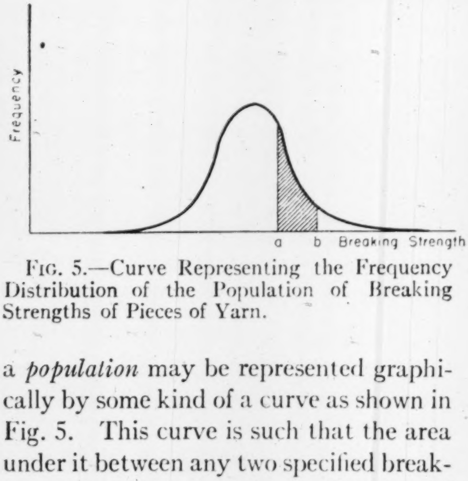


FIG. 5.—Curve Representing the Frequency Distribution of the Population of Breaking Strengths of Pieces of Yarn.

Now it is clear from this example that there is uncertainty in what the breaking strength of any given piece of the yarn will be. This uncertainty is a fundamental characteristic of the yarn, which we have to accept. Under these circumstances about the best thing we can do is to resort to the laws under which the uncertainty operates and make use of them.

A detailed knowledge of the law of breaking strengths for a particular yarn would involve an accurate determination of a curve of the type shown in Fig. 5. Such a curve *would be* the law under which the breaking strengths of pieces of yarn from that bobbin operate. But for practical purposes, the amount of testing would be prohibitive. Therefore the question now arises: How much can we infer about the law of breaking strengths by testing the piece of yarn taken at random? Two pieces? Three pieces? And so on. In other words, how much can we infer about the distribution of breaking strengths in the population by considering random samples of one test, two tests, three tests, and so on? The problem of drawing a random sample of a given number of pieces of yarn from the pieces into which the entire bobbin length can be cut is entirely similar to that of drawing a random sample of articles from a lot, as discussed in section 2(*a*).

Dependence of Knowledge of a Population on Sample Size.—If we take only one piece of yarn *at random* from the bobbin and break it, then the only type of statement we can make *on the basis of the evidence from testing this one piece* is one such as this: The probability is 0.95 that at least 5 per cent of the breaking strengths in the population will exceed the one obtained in the test. This means that if we really knew the distribution of breaking strengths for the population, and could repeat a single-piece test many times, we would find that in about 95 per cent of the tests, at least 5 per cent of the breaking strengths in the population would exceed that obtained in the test. The knowledge we gain about the population of breaking strengths from testing one piece of yarn is therefore very meager.

Now consider two pieces of yarn taken at random from the bobbin. In this case we can say that the probability is 0.95 that at least 22.54 per cent of the population of breaking strengths will exceed the smallest of the two breaking strengths obtained in the test. There is clearly a large gain in knowledge about the population by merely making two tests instead of one.

If 10 pieces taken at random are tested, the probability is 0.95 that at least 74.13 per cent of the population of breaking strengths will exceed the smallest among the 10 breaking strengths obtained.

In general, if n randomly selected pieces are tested, the probability is 0.95 that at least 100 $\sqrt[n]{0.05}$ per cent of the population of breaking strengths will exceed the smallest among the n breaking strengths obtained. We can also say that the probability is 0.95 that at most 100 $\sqrt[n]{0.05}$ per cent of the population of breaking strengths will be less than the largest among the n breaking strengths obtained. These statements are true no matter what shape the distribution curve in Fig. 5 may be.

Curve A, in Fig. 6, shows, for each sample size, at least what per cent of the population values (that is breaking strengths in the yarn example) will exceed the smallest one in the sample (or at least what per cent of the population will be less than the largest one in the sample) with probability 0.95. Similar curves could be drawn for other probabilities such as 0.90 and 0.99 or even 0.50.

Taking curve A as an indication of knowledge about the population of values which can be extracted from samples, it will be seen that the greatest gain obtained by passing from one sample size to the next occurs when one increases the sample size from one to two.

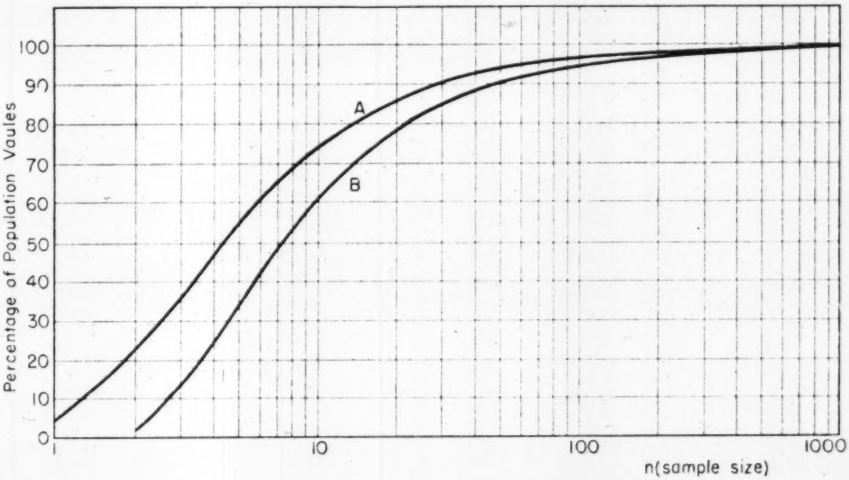


FIG. 6.

Curve A: Percentage of population values exceeding least value (or less than greatest value) in samples with probability of 0.95.
Curve B: Percentage of population values included between least and greatest values in sample with probability of 0.95.

It should be pointed out that the foregoing discussion also applies to samples of measurements from the *population* produced by an operation of making repeated measurements on an object if the sequence of measurements is in a state of statistical control. In particular, if only one measurement is made on an object or a piece of material, very little indeed is learned about it. Two measurements give more knowledge, and so on for more repetitions, the amount of knowledge being shown by curve A in Fig. 6.

Statistically Determined Tolerance Limits.—In dealing with samples of measurements of the type we have been discussing, we may not only be interested in the percentage of the population values exceeding the smallest or less than the largest value in the sample at a given probability level. We may also be interested in the percentage of population values which would lie between the largest and smallest values in the sample at a given probability level. Curve B in Fig. 6 shows, for each sample size, at least what per cent of the population values will fall between the least and greatest values in the sample with probability 0.95. For instance, if we look at $n = 10$, we find the ordinate of curve B to be 0.60. This means that if we make tests on many random samples of 10 pieces of yarn from the same bobbin of yarn and calculate the per cent of the population values falling between the least and greatest values in each group of 10 tests, then in about 95 per cent of the groups of tests the calculated percentages would exceed 60 per cent, the values given by curve B for $n = 10$. If we should adopt the least and greatest values in a sample as statistically determined *tolerance limits*, then curve B essentially shows, for each sample size, the extent to which these tolerance limits will encompass the population of values at the 0.95 probability level. One could also show curves similar to B for the case in which the second smallest and second largest values, third smallest and third largest values and other *order statistics* are used for the statistically determined tolerance limits. For further details see the paper by Murphy (**11**).

3. VARIABILITY OF SAMPLE AVERAGES FROM A BULK

Sampling Law of Sample Averages:

In sampling problems of the type discussed in sections 2(*a*) and (*b*), we are often interested in the law of uncertainty of averages (arithmetic means) of samples, that is, the extent to which samples averages are subject to fluctuations from one sample to another. We are not interested in these laws purely for their own sake; we are interested in making some kind of a statement about the average of the population of individual values or measurements by using the information embodied in the sample.

If we take a large number of random samples of n tests and average the n measurements obtained in each sample, then these averages will themselves form a population of averages (which is not to be confused with the population of individual measurements) which will have a distribution curve which can be closely approximated by the well-known symmetrical bell-shaped curve known as the *normal* curve. This will be true for almost any kind of a distribution curve for the population of individual measurements (as shown in Fig. 5) which may occur in practical situations. The population of sample averages will have the same average as the population of individual measurements, and this average is *unknown* in practice. For the purposes of this paper we do not need to discuss this normal distribution law further.

Confidence Limits for the Average of a Population.—We can put the preceding information together so that when a sample of measurements is obtained we can make a statement that the *unknown* average of the population of individual measurements will be included between two specified limits with a given probability. Such limits are called *confidence limits.*

To make the procedure for establishing confidence limits of the average of a population of individual measurements more precise, suppose we have a random sample of n measurements, and let the measurements be denoted by X_1, X_2, $X_3, \ldots, X_n$. We calculate two quantities from these measurements, namely, the average $\bar{X}$ and the standard deviation s,* defined as follows:

$$\bar{X} = \frac{1}{n}(X_1 + X_2 + \cdots + X_n)$$

$$s = \sqrt{\frac{(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \cdots + (X_n - \bar{X})^2}{(n-1)}}$$

If we adopt a 95 per cent *level of confidence,* the statement which we can make about the average of the population of individual measurements is this:

The probability is approximately 0.95 *that the average of the population of individual measurements is included between the* 95 *per cent confidence limits*

$$\bar{X} \pm t_{0.05}\frac{s}{\sqrt{n}}$$

where $t_{0.05}$ *depends on* n, *the sample size.* For instance, the values of $t_{0.05}$ for n = 2, 5, 10, 20, and 30 are 12.706, 2.776, 2.262, 2.093, and 2.045 respectively. Other values are given in (**12**).[3]

The correct statistical interpretation of these 95 per cent confidence limits is this: Suppose many random samples of size n are drawn and that the confidence limits $\bar{X} \pm t_{0.05}\frac{s}{\sqrt{n}}$ are calculated for each sample, then in about 95 per cent of the samples it will be found that the confidence limits include the average of the population between them.

The precise form of this statement should be noted very carefully. It is widely misunderstood and misused. In particular, it should be observed that the statement *does not mean* that about 95 per cent of the individual measurements in the population will be included between the limits $\bar{X} \pm t_{0.05}\frac{s}{\sqrt{n}}$, yet this is the interpretation often given to it.

As a simple illustrative example, suppose that for a random sample of 10 breaking strengths of yarn of a certain type from a bobbin we find

$$\bar{X} = 52.0 \text{ oz.}$$
$$s = 6.3 \text{ oz.}$$

The 95 per cent confidence limits for the average breaking strength of the bobbin of yarn (that is, of the population of individual measurements of breaking strengths from which this random sample is assumed to have been drawn) are

$$52.0 \pm (2.262)\frac{6.3}{\sqrt{10}} \text{ oz.}$$

or

$$52.0 \pm 5.0 \text{ oz.}$$

When we obtain these limits we simply make the statement that on the basis of the sample evidence the average breaking strength of the bobbin of yarn is between 52.0 + 5.0 oz. and 52.0 − 5.0 oz. with the understanding that if we were to repeat the sampling many times, calculating limits each time, and making such a statement, then only about 95 per cent of the statements would be correct. That is, in only about 95 per cent of the samples will the calculated confidence limits include the average breaking strength of the bobbin of yarn.

It should be emphasized that we could set up confidence limits at the 0.90 or 0.99 or any other probability level. The only changes that would be made would be in the factor $t_{0.05}$.

---

* Here we are dealing with the standard deviation of a sample of measurements and we use the now commonly used symbol s, rather than σ.

[3] Values for obtaining 90 and 99 per cent confidence limits of the average of the population are also given in Table II of Supplement A of the A.S.T.M. Manual on Presentation of Data. (Issued as separate publication, *STP No. 15-B.*)

4. REPRESENTATIVE AND COMPOSITE SAMPLING

Ideal versus Practical Conditions of Sampling:

The laws of sampling variation for Stage I sampling are applicable under certain "ideal" conditions. To recapitulate: The conditions under which the laws of sampling from a lot discussed in section 2(*a*) apply require that all pieces in the lot have "equal chances" of being included in the sample. The conditons under which the sampling laws discussed in sections 2(*b*) and 3 apply require that the bulk being sampled be considered as being made up of a large number of *sampling units* and that in drawing a sample of n of these units, all sampling units have "equal chances" of being included in the sample.

It is well known that in practical situations these "ideal" conditions are usually only partially realized at best. As explained in section 2(*a*), it is often impractical to set up a mechanically randomized method for selecting a sample from a lot of articles. A compromise between a mechanical method and human selection usually has to be adopted, and such a method of selection is always subject to the frailties of human choice. The ultimate validation of the applicability of sampling laws of the kind discussed in section 2(*a*) for such a compromise has to be done experimentally. Except in certain simple instances, it would be very difficult or indeed impossible to determine what alterations would be needed in the mathematical laws to fit such compromise methods of sampling. In case it is impossible to draw a random sample from the lot, the question arises as to whether there are general principles which can be followed which would still permit us to use the "ideal" sampling laws as some kind of a guide. One partial answer to this question is the use of *representative sampling*, which will be discussed presently.

Similar statements apply to problems of sampling from a bulk of material. As in sampling from a lot of product pieces, the question arises as to whether there are principles which can be followed which will still allow us to be guided by sampling laws. Again, one answer is to make use of *representative sampling.*

Representative sampling.—We have mentioned representative sampling as a method of overcoming difficulties presented when random sampling of lots of product pieces or lots of bulk material is not feasible. Under such circumstances the only type of procedure one can safely use is one which will keep the fraction of defectives in the sample, when averaged over many samples, the same as the unknown fraction of defectives in the lot. The method of *representative* or *stratified* sampling is such a procedure. By this method of sampling a lot, we mean a method which consists of subdividing the lot into parts or sublots and drawing at random from each sublot, the number of pieces or units to be drawn from the various sublots to be as nearly proportional to numbers of pieces in the sublots as possible. The operating characteristic curve for any sampling plan based on this method of sampling would depend on how much variability there is from sublot to sublot in comparison to that within each sublot, but in practice the curve for representative sampling would not be very different from that of random sampling. Effective use of representative sampling depends on knowledge of how to break a lot into sublots so that the variation within lots is relatively small and that between lots relatively large. Of course, in many situations lots are already essentially divided into sublots.

For example, a lot of vacuum tubes may consist of three pairs of large cartons of equal size. A representative sample of 12 tubes might consist of 2 tubes taken at random from each carton. Or it might consist of three sets of four tubes, each set of four being drawn at random from one carton taken at random from each pair.

In the case of sampling from a lot of bulk material, representative sampling consists of dividing the material into sublots and taking a number of sampling units from each sublot, the number of units being as nearly as possible proportional to the volume of bulk material in each sublot. As in the case of lots of product pieces, sublots are often determined by manufacturing, shipping, storing, and other conditions.

As in the case of lots of product pieces, the effectiveness of representative sampling depends on how much variability there is from one sublot to another as contrasted to the variability within each sublot.

If representative sampling of bulk material is carried out effectively, then the results of such samples as compared with those of random samples lead us, when applying the sampling laws of section 2, to make conservative statements about the population of measurements from which the samples are drawn. Let us illustrate by considering an example. It will be remembered from section 2(*a*) that if we take a random sample of 10 pieces of yarn and obtain their breaking strengths, then the probability is *equal to* 0.95 that at least 74.13 per cent of the population values would exceed the smallest breaking strength in the sample. In representative sampling, the probability of this occurring is *at least* 0.95. How much greater than 0.95 this probability would be depends on how large the variability is from sublot to sublot as compared with the variability within each sublot.

Representative sampling as compared with random sampling from a bulk of material also permits conservative statements about confidence limits for the average of the population of individual measurements. Referring to section 3, we may say that if a representative sample had been used the probability that $\bar{X} \pm t_{0.05} \frac{s}{\sqrt{n}}$ includes the population average will be *at least* equal to the probability of this happening if a random sample had been used.

Composite samples.—In many situations, the sampling units obtained in representative sampling of a lot of bulk material are thoroughly mixed together into a *composite sample.* Composite sampling is a device which, if carried out properly, approximates taking a random sample of one unit from the lot if the material in the lot were thoroughly mixed. The degree of approximation depends on the thoroughness of mixing within each sublot—the more thorough the mixing the closer the approximation.

Composite sampling clearly forfeits all information regarding variability from one sublot to another. This, of course, is unimportant only if the material is sooner or later to be thoroughly mixed, or, more generally, if knowledge of variability from one part of the lot to another is irrelevant. If it is important to have information about various parts of a lot of bulk material, then clearly composite samples cannot be used.

5. BIASES AND OTHER ASPECTS OF SAMPLING

The main function of Stage I sampling is to make inferences about the lot from which the sample is drawn. In making inferences we consider what would happen if sampling is repeated a large number of times and then introduce the idea of a population of values obtained by repeated sampling. Sampling laws enable us to learn something about this population. The vehicles by which the inferences are made are therefore sampling laws and the concept of a population of values. The accuracy of our inferences depends on how carefully the sampling is carried out.

Bias in Sampling.—Representative sampling has been discussed as a device for diminishing the effect of nonuniformity from one part of a lot to another, and hence to lower the danger of bias due to inability to sample nonuniform material at random. But there are other pitfalls and obstacles than nonuniformity within lots which beset the sampling expert. Many of them are traps into which he unwittingly allows himself to be caught. Every field in which sampling is applied has its own peculiar traps. To try to discuss all of the things that can cause bias in sampling is like trying to discuss all of the ways a law can be can be violated. But it might be useful to mention a few of the more glaring violations of good sampling practice.

One of the most important sources of bias in a situation involving sampling from a bulk of material is the effect of the size of sampling units on the characteristic being tested. The main danger occurs when the sampling units are chosen too small. For instance in cotton sampling, if sample units are taken as small tufts that can be pulled from the bale between the fingers, such a sample will be biased as far as length of fiber is concerned. The sample will contain too many long fibers. In sampling lump coal sampling units which are too small tend to over-sample the smaller particles of coal. The size of the sampling unit should be large enough to avoid bias in selecting material from the lot. If it is too large to be used in making a test, particularly chemical, then the sampling unit actually selected has to be processed by grinding, pulverizing, etc., so that it becomes homogeneous before a portion is selected for actual test. In the example of cotton sampling, a larger unit than a pinched tuft is necessary to avoid bias in length of fiber. If only a small portion is needed for a fiber length test, a method of successively subdividing a combed form of the sample has to be used.

Another very common source of bias is the unwarranted rejection of tests resulting in measurements which "look" too high or too low and basing conclusions on the remaining values, or allowing retests to replace the rejected tests. In some cases, of course, something obviously goes wrong to invalidate a test, but whenever possible it should be decided that something went wrong *before* looking at the final measurements. If a decision is made to reject on the basis of test results, there usually remains some question as to whether the very high or very low measurement should not be considered as a sampling fluctuation. If it is really a sampling fluctuation and is rejected, the conclusions are subject to considerable bias, particularly if the number of tests (sample size) is very small.

REFERENCES

(1) A. A. Michelson, F. G. Pease, and F. Pearson, "Measurement of the Velocity of Light in a Partial Vacuum," *Astrophysical Journal,* Vol. 82, pp. 26–61 (1935).

(2) W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., New York, N. Y. (1931).

(3) W. A. Shewhart, "Statistical Method from the Viewpoint of Quality Control," The Graduate School, The Department of Agriculture, Washington (1939).

(4) "Control Chart Method of Controlling Quality During Production," Am. Standards Assn., New York, N. Y. (1942).

(5) P. S. Olmstead, "Distribution of Sample Arrangements for Runs Up and Down," *Annals of Mathematical Statistics*, Vol. 17, pp. 24–33 (1946).

(6) R. L. Anderson, "Distribution of the Serial Correlation Coefficient," *Annals of Mathematical Statistics*, Vol. 13, pp. 1–13 (1942).

(7) K. A. Brownlee, "*Industrial Experimentation*," Chemical Publishing Co., New York, N. Y. (1947).

(8) Harold F. Dodge and Harry G. Romig, "Sampling Inspection Tables," John Wiley and Sons, Inc., New York, N. Y. (1944).

(9) "Standard Sampling Inspection Tables for Inspection by Attributes," *Appendix X, General Specifications for Inspection of Material*, Navy Department (1946).

(10) Statistical Research Group, Columbia University, "Sampling Inspection," McGraw-Hill Book Co., New York, N. Y. (1948).

(11) R. B. Murphy, "Non-Parametric Tolerance Limits," *Annals of Mathematical Statistics*, Vol. 19 (1948).

(12) R. A. Fisher, "Statistical Methods for Research Workers," Oliver and Boyd, Edinburgh and London (1946).

DISCUSSION

Mr. Robert H. Lace.[1]—If you must have a decision as to the quality of the single bobbin of yarn and you have to make the decision on the basis of a very small number of samples because of the destructive nature of the test, it is rather uncomfortable, to say the least, to make a statement that your results are going to be only 5 and 4 per cent accurate.

Mr. S. S. Wilks (*author*).—I tried to underline, when I made the statement, that if you use only the evidence that you have on that one test from the bobbin and make use of no other knowledge, that is correct. If you do make a statement which implies greater knowledge about the breaking strength of the yarn and analyze the statement very carefully you will probably find that, even though it cannot be stated very precisely, the statement really depends on earlier knowledge of the yarn or similar yarn. Perhaps you have even had a quality-control chart on the yarn made under the given process. If so you certainly have more knowledge about the yarn than that provided by a single isolated test.

However, suppose you are going into something completely new, where you do not know the universe, given by the curve we talked about in Fig. 5. Then if you base your statement on only the evidence that is given by testing pieces from the universe, you would have the kind of statements we can make from Fig. 6. These statements contain considerable uncertainty; but that is how uncertain the results of sampling are.

Mr. Lace.—But I think that out in the field particularly, you may not know the past history (you would not if you happen to be a consumer). If you do not know the past quality, do you not run a tremendous risk if you do not know the accuracy of the sample? You have to make a decision as to what you expect to get from production on the basis of the sample, and information that is only 4 per cent accurate will not do it.

Mr. Wilks.—Well, if you make only one test then if you use only the information of that test, the amount of knowledge you get out of the one test is very small.

Mr. Lace.—Frequently the nature of the test is such that you cannot go from 1 to 4 samples with any hope of increased accuracy.

Mr. Wilks.—When you go from 1 to 4, the knowledge steps up very sharply if no previous knowledge is utilized. The steepness of curve A in Fig. 6 shows the gain you get in knowledge in going to larger samples.

---

[1] Quality Control Manager, Ditto, Inc., Chicago, Ill

ON VARIATION IN MATERIALS, TESTING, AND SAMPLE SIZES*

By Leslie E. Simon[1]

Synopsis

The scientific sampling of materials is regarded as consisting of three essential parts: (1) Design of Experiment, (2) Execution of Tests, and (3) Interpretation of Results.

The *Design of Experiment* involves (*a*) careful identification of the test phenomena, (*b*) evidence of state of predictability in material sampled, and (*c*) a technical procedure designed to yield a maximum of information with a minimum of cost and effort.

The *Execution* phase must contemplate (*a*) the variability in the material and (*b*) precision and accuracy of measuring processes.

The *Interpretation of Results* should be recognized (*a*) as a predictive process (*b*) as being sensitive to both the design of experiment and statistical control of its execution and (*c*) as dependent upon an evaluation of these relations for a valid statement about the precision and accuracy of prediction.

In the *Use of Quality Control* little or no change need be made in usual terminology; the change consists of making use of probability information for calculated risks so as to minimize such costs as the cost of inspection plus the cost of consequences of accepting sub-standard materials, etc., thereby resulting in an important economy in over-all effort.

Current interest in scientific techniques for sampling and testing brings with it both interesting applications and misapplications. According to the *New York Times* of February 10, 1948, English Minister of Health Aneurin Bevan recently related to the House of Commons an application by distinguished men, if not a distinguished application.

"G. K. Chesterton and Hilaire Belloc had been arguing as to the cause of drunkenness, and they decided to apply the scientific method. One night they drank whiskey and water, and got drunk. The next night they drank brandy and water, and got drunk again. On the third night they drank gin and water, with the same result. Since water was the constant factor in all three trials, they concluded that water was responsible for their drunkenness."

Most of us would not criticize their design of experiment—perhaps there is much to recommend it—but we feel that in our testing of materials we would scarcely be guilty of such spurious correlation and mal-interpretation of results. However, we might well consider how many almost equally serious errors we commit from day to day because of not clearly defining our aims and purposes, because of not properly identifying the kind of variation of material under test and because of not recognizing the effect of sample size.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Colonel, Ordnance Dept., Director, Ballistic Research Labs., Aberdeen Proving Ground, Md.

SAMPLING—A PREDICTIVE PROCESS

Most of us are busy day in and day out in some specialized field of work, handling materials, facts, and figures. Many of us feel that we know pretty well what we are doing, and that we do not take chances, or at least, not many. Those who feel that way are generally fair-minded about inviting anyone to show them, if they are wrong—especially if the new viewpoint is beneficial—so I am going to discuss for those who are open to conviction a challenging question. Do we not know very little about the real quality of materials? Do we not accept as facts things that are largely mere predictions and are not usual figures on quality of materials little more than estimates with no known degree of certainty?

First, let me score the point about prediction. Suppose that you take a single sample from a consignment of 2-in. bolts. You measure it with a desk rule and say, "Yes, it is two inches." Another person may pick it up, measure it with a machinist's scale, and say, "No, it is only 1.96 inches." To settle the point, you might call in an inspector with a precision micrometer, have him measure it 10 times, and take the mean. His mean value might be 1.965. If we were to set up a sine bar, we would get another answer; if the physicist measured it, using the wave length of monochromatic light, another. The point is that we can never know the absolute length of the bolt, and that what we really mean when we state that the length is 1.95 in. is that we *predict* that if a large number of measurements were taken by a skilled operator, with a well calibrated and appropriate instrument, the mean of the measurements would approach 1.95 in. and not some significantly different value.

The element of prediction may be trivial in the specification of length of a bolt, but the uncertainty of prediction is not trivial in testing the corrosiveness of oil, where one must carefully specify how a copper test plate will be freshly polished, how the oil will be applied, the temperature, and the judgment of corrosion. The important thing is knowing something about the degree of uncertainty so that the method of measurement can be specified in such a way that the element of prediction *will be* trivial. In this connection, the Committee on Quality Control of Materials is trying to furnish services to the technical committees that write the A.S.T.M. specifications that will enable them to decide wisely in the many less obvious cases that lie between the length of a bolt and the corrosiveness of an oil.

However, the facts and figures based on the prediction of the quality of samples may not be our most hazardous predictions. Why do we take samples at all? Certainly the few measurements on the samples are not in themselves of any practical consequence. We take samples for one reason only: in order to predict the quality of the remaining aggregation from which we draw the samples. Furthermore, we know that material varies. If we take a sample of two items from a consignment and test them, the two test results will generally be different. If we begin to consider carefully what we are going to say about the unsampled items, we begin to feel a bit "sheepish." We are perfectly sure that if we tested all the items, they also would be not only different but would not even fall within the limits of the two test results. It is evident that before we can make valid predictions, we need to know something about the *variability* of the material; that is, the distribution of the quality characteristic in the lot of items.

Just in case that life does not look uncertain enough at this point, let me say something much worse. Unless there is a relationship—preferably a known relationship—between sample and unsampled remainder (homogeneity within the lot), an inference of lot quality from sample quality is impossible. Very frequently there is no evidence of a relationship at all between sample and lot.

In summary, a sample of one tells us that *before* the sample was taken, one item in the lot had such-and-such value, and that is about all. A sample of two (the test results being different) tells us that there is some variation in the lot (or in the measurements) but indicates little about the degree of variation. As the sample size is increased, our ability to predict the limits of variation increases, provided there is a relationship between sample and unsampled remainder. Thus it is evident that the testing of materials should involve at least three steps: (1) design of experiment, (2) execution, and (3) interpretation of results.

DESIGN OF EXPERIMENT

The first item in the design of experiment[2] is the careful identification of the important test phenomena, lest testing be done on a characteristic which is only poorly related to engineering performance. It seems trite to say, "know what you are testing for," but that is a point on which many material tests fail. I have heard steel men say that one of the most significant contributions of the war was the specification for gun-tube forgings. Yet, the principal fault of the old specification was not necessarily statistical—the fault was in design of experiment. The reasons for the difficulties in sampling are obvious. Sampling a gun-tube destroys it, so samples must be taken from both ends of the finished and heat-treated forging just outside the metal required for the finished, machined tube. This practice was changed by adaptation of more systematic methods of sampling with provision for statistical consideration of results and provision to have specimens taken from the mid-length of check forgings only when impact testing is required. In addition to the above, a comprehensive metallurgical study assisted by statistical analyses did indicate that a considerable reduction in testing might be effected without loss in quality. Further, such analyses assisted materially in keeping quality in control. The knowledge contributed both to better steel making and to more economical use of materials; and towards the end of the war, the Ordnance Department was making thin-skinned guns that literally swelled when the projectile passed through them like the ostrich's neck when he swallows an orange.

PREDICTABILITY

If the sampler has access to the continous flow of product from the manufacturer, not only does the verification of a predictable relationship between sample and lot become easy,[3] but great economy in sampling becomes practicable, because a background of past knowledge can be combined with the knowledge from the specific sample. Under these conditions, the sample can be used for the sole purpose of indicating no evidence of a change from previously established quality. A manufacturer who keeps quality control records as described in the A.S.T.M. Manual on Presentation of Data[4] can furnish this essential information to a consumer. However, in the absence of access to quality control records, the process of verifying a state of statistical predictability becomes prohibitively expensive,[5] and it is common practice to place reliance on the reputation of the manufacturer; on all the products being from the same heat of steel; on all articles being from the same industrial lot; and similar assurances of homogeneity.

---

[2] R. A. Fisher, "The Design of Experiments," Haffner Publishing Co., New York, N. Y.

[3] L. E. Simon, "The Industrial Lot and Its Samping Implications," *Journal of the Franklin Institute*, Vol. 237, No. 5, May, 1944.

[4] A.S.T.M. Manual on Presentation of Data, Am. Soc. Testing Mats. (Issued as separate publication, *STP No 15-B*.)

It often happens that there are engineering reasons for suspecting lack of homogeneity, and provision is made for taking samples from the top, middle, and bottom; from every batch; from every box, etc. If variation in materials is by strata and if the stratification can be identified *a priori*, great economies can be made in sampling and testing. Without going into the subject exhaustively, there is one point in technical procedure which is of such outstanding importance that it deserves special mention. Sampling of lots, batches, or other categories is often prescribed on a percentage basis. This is a grievous error. The evidence produced by a sample is proportional to the sample size and almost independent of the size of lot from which the sample is drawn.

Let me put that idea in numbers. A sample of 10 from a lot of 100 is only a slightly more reliable witness of lot quality than a sample of 10 from a lot of 1000. If sampling were by attributes it would take a sample of only 11 from the lot of 1000 to yield precision slightly better than that of the sample of 10 from a lot of 100. Thus, for once, for all, we should make an end of writing provisions for taking a sample of *p* per cent of the lot. It is not only waste but often results in preferential treatment of poorer producers. The use of statistical methods in the design of experiment will provide many powerful economies of this sort.

EXECUTION OF EXPERIMENT

The design of the sampling experiment must anticipate the variations introduced by the execution phase of the experiment. When an engineer prescribes a minimum average of 50,000 psi. on a small sample to meet a real requirement of 30,000 psi. on the part of the lot, he generally feels that he is doing so because of anticipated variation in materials. However, only a part of that anticipated variation is in the materials. A part of the variation is due to the incertitude associated with the small sample. A part is due to the method of selection of the sample, which may be far from the wisest as was the case in the gun-forging specifications. A part is due to variability in the measuring process, including both errors of the instrument and errors of the operator. If cognizance is not taken of the variations introduced by execution of experiment, materials may be accepted which are unsatisfactory and also satisfactory materials may be rejected, not because of their variability but because of variability in the method of measurement.

There are three well-known attacks on the problem of variations introduced by execution of experiment. The engineer's usual attack consists of some use of standards or working standards (to minimize errors of calibration) plus reliance on factors of safety. The physicist tends simply to over-instrumentation; for example, if he needs to measure time to 0.01 sec., and his electric clock is doubtful at that figure, he will employ an electronic counter-chronograph good to 0.00001 sec. Techniques which would be extravagantly prohibitive for repetitive industrial measurements may be an economic way for the physicist to dispense with the whole problem of variations of measurement. The Quality Control Engineer, however, carries an additional burden. He must seek the economic use of materials. Consequently, he must attempt to strike an economic balance between the costs of inspection and the costs of the consequences of making errors either by occasionally accepting sub-standard materials or occasionally rejecting materials that are standard or better. Enormous savings are possible for producer, consumer, and the public through the recognition and use of the methods of the Quality Control Engineer.

---

[5] For quality characteristics tested on an attribute basis; that is, go, no-go; defective, non-defective; and so forth, mere random selection of sample insures validity of prediction from sample to unique lot.

INTERPRETATION OF RESULTS

In order to achieve the economic advantages of the statistical viewpoint, not only must one recognize the predictive nature of all sampling processes, but be able to evaluate, at least approximately, the probabilities associated with the predictions. Action (acceptance, further inspection, re-working, re-grading, or rejection) must be predicated upon an evaluation of probable sampling information together with other available knowledge.

For example, if the variability of a material in terms of its standard deviation, σ, is known, and if its average based on a sample of n specimens is A, then the material can be safely used where a minimum average value B is required, provided that $A - B$ is greater than 3σ divided by the $\sqrt{n}$. If a risk of one chance in ten of failure can be accepted (say that the defective material will be discovered in assembly and results in only a small cost for correction), $A - B$ greater than about 1.3σ divided by the $\sqrt{n}$ is the appropriate criterion.

USE OF QUALITY CONTROL

Many similar provisions for the economic achievement of one's aim in a known and calculated way are available for the scientific specification of quality. The textual matter need say nothing specifically about design of experiment, execution, and interpretation of results any more than the scientist defines his method of hypothesis, experiment, and test of hypothesis. It is not necessary to employ the language of statistics. In fact, only the trained statistician may be able to detect the procedures based on economic and calculated risks from those based on intuitive estimates, usual practices, and guesses. However, there is a great deal of difference in the thinking that goes into the two kinds of procedures.

Is it too much to expect the technical committees of the A.S.T.M. to use some of the methods of statistical quality control in specifying the quality of materials? The Society's Committee E-11 on Quality Control of Materials does not think that it is. This committee is revising the A.S.T.M. Manual on Presentation of Data and trying to arrange a comprehensive treatment of quality control of materials in such a way that the engineer can find conveniently the information that he wants, expressed largely in terms with which he is familiar. Furthermore, the membership of Committee E-11 is available to render assistance in difficult A.S.T.M. problems.

I believe that the engineers of A.S.T.M. are forced by circumstances consciously or unconsciously to make a choice between two alternatives. Either they will identify themselves with the cynically defined practical man—the man who is either too busy or not sufficiently alert to find out what a new thing is all about—and the science of engineering will tend to lag behind other sciences or else they will learn to use the new and powerful engineering tools now provided by science and lead engineering to new economic triumphs. Since the A.S.T.M. was one of the pioneers of Quality Control, there is little doubt about your meeting the challenge.

DISCUSSION

MR. J. C. KNAPP.[1]—I should like to ask Mr. Simon about how many years he thinks it will be before engineers will take advantage of the information available from quality-control methods?

MR. LESLIE E. SIMON (*author*).—A formal prediction is impracticable, and I think that any prediction is highly problematical. The greatest stimulus to the currently broad use of quality-control methods came out of the war. Quality control prior to the war was confined principally to large corporations that could afford good engineering staffs. You found it used in places like the Bell Telephone Laboratories, the large electric companies, General Electric, Westinghouse Electric, and so forth. During the war we got an enormous stimulus to quality control in an indirect way. Instead of trying to accept munitions on a quality-control basis, which probably would have been politically infeasible, we used sampling techniques (largely known today as single and double sampling) in which Mr. Gause and Mr. Dodge both did leading work.

What we did was place the acceptance of materials on a scientific basis that made all the manufacturers feel that they were getting a square deal and yet feel that they were being checked up on very carefully. When a manufacturer began to get rejections he would ask why his competitor was getting by and he was not. You would often find that the competitor was using quality-control methods. That gave a big stimulus to quality control.

I do not know what can give another stimulus to quality control such as the war, but I hope we shall not have to resort to another war for that purpose. On the other hand, I have found a large number of colleges today are giving statistical methods under various guises. Some of them are known as industrial statistics and some give a kind of mixture between engineering statistics and business statistics.

I think the American Society for Engineering Education is going to support quality control, because it is felt that if we do not have statistics in engineering that engineering will slip behind. Biology and the social sciences have forged ahead by using statistical methods, and the engineer is in a similar position. The engineer often cannot do much about his mistakes if he samples wrong; the material has already gone down the production line and been processed. The people in the social sciences cannot re-sample, either. They take certain samples, and if they go back to take another sample conditions will have changed. So there are similarities between process engineering and the social sciences which are recognized by the leading teachers of engineering.

I believe the biggest hope in the broad propagation, of quality control lies in the colleges. One must realize that the engineer of the old school is a bit slow about accepting these techniques and somewhat critical of the young engineer who steps forward and uses them. However, the young engineers are going to step forward; they are successful and they are sure to be recognized.

---

[1] Superintendent of Quality Control, Ford Motor Company of Canada, Windsor, Ontario, Canada.

MR. O. P. BECKWITH.[2]—I wonder whether Mr. Simon would say a few words on the growth of statistical quality control concepts in specifications of Federal procurement agencies? I think we have an interest in that, because, after all, they are competitors, you might say, of the A.S.T.M., in that they too are producers of standards. I think it should be of interest to all of us to know if the Federal procurement agencies are greatly extending statistical quality control qualifications in their specifications because it would seem to indicate an advance in their thinking over the thinking of people in the A.S.T.M.

MR. SIMON.—I would say there has been growth, because 15 yr. ago it was zero. Statistical methods have been put into specifications and in general are not detectible. It is not necessary that the statistics be obvious. One can write a specification in such a way that in reading it one cannot readily detect whether it was predicated on statistical concepts or on ordinary engineering concepts. It would take a trained statistician to detect the difference. It does make them complicated.

We have a number of them that were written before the war, when I was more active in that field. I understand now that there is a growing movement on, particularly sponsored by the Navy, to make all of the specifications that way.

Mr. Hollady, from the Ordnance Office, the Research and Materials Division, who is much more familiar than I with what is happening currently may wish to comment.

MR. E. L. HOLLADY.[3]—I corroborate what Mr. Simon has said. There was no definite plan for incorporating statistical methods of sampling and acceptance before the war, with the possible exception of specifications coming out of a few agencies having to do with ammunition.

At the present time there is a definite trend towards the adoption of more systematic methods of sampling and consideration of the results of tests.

The Federal Specifications Board Technical Committee on Metals currently is working on new specifications for carbon steel bars and alloy steel bars. They probably will be the most widely used individual specifications in the whole Federal government.

We have had recommendations from some agencies reviewing the preliminary drafts that we adopt a plan of statistical sampling and consideration of the results of tests. The first proposals submitted back to the steel industry representatives were not acceptable, largely or primarily because they greatly increased the amount of testing over that normally considered practical. We are referring the matter to some other agencies to review carefully, to see if it is possible to benefit from a more systematic method of sampling without greatly increasing the amount of testing.

The first recommendations we recieved were from people who were quite familiar with statistical methods of sampling and quality control but not thoroughly familiar with the particular problems of procurement of steel products. Therefore, we will have to apply some of the principles indicated by Mr. Simon's paper, that is, use all of the available knowledge regarding the product, its source, the processes applied, its production, knowledge of how carefully those were controlled, with the hope that we can then get a method of sampling that will give us better assurance than we have had, without unduly increasing the amount of testing.

I definitely feel that the more systematic methods are going to be reflected in an even greater degree in the specifications of the Federal Government as the specifications writers and engineers become more familiar with what can be accomplished. I feel that there is no conflict between good common horse sense in specifications and the application of these statistical methods. The thing that has scared most of our people is the fact that the first sample they saw was something elaborate superimposed on an old specification. I think both Mr. Simon and Mr. Gause are thoroughly familiar with the problem we have in that respect—at least in Ordnance—and will agree with me that we can apply statistical methods practically throughout the specifications system.

---

[2] Chief, Product Engineering Lab., Alexander Smith and Sons Carpet Co., Yonkers, N. Y.

[3] Chief, Materials Section, Office Chief of Ordnance, Department of the Army, Washington, D. C

CHAIRMAN H. F. DODGE.[4]—Mr. Holladay, may I ask how you view A.S.T.M. specifications in this regard, in general? I refer particularly to the sampling plans and inspection procedures contained in A.S.T.M. specifications. Are they considered adequate and satisfactory?

MR. HOLLADY.—I think there is room for improvement in the A.S.T.M. specifications, just as there is room for improvement in the government specifications. We all have been inclined in the past to take the easiest way out and say one sample from each lot, and at times not be too definite as to what constitutes a lot. So I think there is room for improvement in practically every A.S.T.M. material specification by applying statistical methods and through thorough consideration of the significant factors. In many cases we are inspecting and testing for properties and characteristics which are not really significant in the use of the product involved. They happen to be things which are easily determined or have always been determined.

I believe that the A.S.T.M. committees as well as our own in the Government should become better acquainted with what can be accomplished. Little, if any, good can be done by going out and occasionally bringing in a statistician and saying, "Now, you sit down and quickly apply a new plan to this specification." We who are working with specifications either must become more statistically minded or bring into our committees and groups individuals with good training in statistical methods who, after working with us to become familiar with the peculiar problems, can improve our specifications.

MR. F. B. SHAW.[5]—Two questions have been raised concerning the use of statistical sampling in Federal Government specifications. In answering these questions, Mr. Simon remarked that it is difficult to tell from reading a specification whether a statistical sampling method has been applied or not. Mr. Holladv discussed that question as it applies to Ordnance. The practice is a little bit different with the Chemical Corps.

As those who are producers for the Chemical Corps know, we have attempted to put a statistical sampling quality-control procedure in every pertinent specification we have written since the middle of 1945. When you read one of our specifications you can readily tell whether that has been accomplished or not by referring to that paragraph in the "F" section which covers sampling.

Since it is not very practical to put a complete set of sampling tables in each specification, our inspectors furnish such tables. As a result, the sampling paragraph of the "F" section of each specification incorporating a statistical sampling plan reads essentially as follows: "Sampling and inspection shall be conducted in accordance with authorized classification of defects and sampling tables in effect at the time of sampling. Whenever acceptability is based on the inspection of a sample, failure of the sample to meet the specified requirements shall be cause for rejection of the lot which the sample represents."

---

[4] Quality Results Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

[5] Chief, Engineering Div., Chemical Corps, Technical Command, Army Chemical Center, Md.

MR. S. H. KATZ.[6]—I might add that the Chemical Corps has become statistical minded. This started during the war and has been evident ever since. A group within the service now receives training in statistics as applied to testing under specifications, and to quality control, as well as the broader applications.

MR. ARTHUR W. CARPENTER.[7]—I should like to suggest that there is one factor in addition to those mentioned by Mr. Simon that is going to be very important in stimulating the growth and application of statistical quality control.

As most manufacturers' representatives who are here this morning realize, we are entering a period of severe industrial competition, which will certainly become one of quality. During the next few years, the manufacturer who does not stay alert and utilize the methods that are now being made available to him for control of quality and elimination of waste is going to be hopelessly behind the procession. It is a fact that initially applications of statistical quality control usually involve considerable expenditures, both for personnel and for organization training. In general, manufacturers find that their sampling increases by an amount that makes them gasp. Ultimately, however, the savings through reduction in defective material or product which would otherwise have involved waste will usually more than offset the increased expense. In addition, there will be intangible gains from improved customer protection and satisfaction. I feel sure that future suppliers will encounter increasing demand from their customers for this type of quality protection.

I have in mind a recent case where application of statistical quality control in production of a small automotive part involved an increased expenditure of $3000 a week in the manufacture of that particular product. In these days of competition, such increases, of course, come under closest scrutiny. Within a month the cost had dropped $4000 a week as a result of a reduction of the per cent defective of the out-going product from more than 3½ per cent to less than 1 per cent. And not only was there a reduction in cost, but the customer doubled the quantity on order and complimented the manufacturer on the enormous improvement that was being made in the quality of goods being delivered.

Now, this is not a testimonial meeting, but if it has brought about a realization of what statistical quality control can do and what is back of all of this very important operative technique that is being outlined, this symposium will certainly have accomplished its purpose.

---

[6] Consultant, Chemical Corps Technical Command, Army Chemical Center, Md.

[7] Manager of Testing Labs., The B. F. Goodrich Co., Akron, Ohio.

THE AMOUNT OF INSPECTION AS A FUNCTION OF CONTROL OF QUALITY[*]

By G. R. Gause[1]

Synopsis

One of the important developments in the field of quality control is the use of sampling procedures wherein the amount of inspection or sample size depends upon the extent to which quality of product is satisfactorily controlled. Under these procedures, inspection results obtained on samples from successive lots of product are summarized to obtain a measure of the general level of quality and its uniformity from lot to lot. Whenever such summaries indicate a satisfactory state of control reductions in the amount of inspection can safely be made. Various techniques for applying these principles are described and their application under different conditions discussed.

Inspection, like any other production or procurement function, costs money and consumes manpower. It is therefore essential that unnecessary inspections be avoided and that available inspection effort be directed toward those points where it is most needed. The decision as to how much inspection to carry out on a particular product at a particular stage of production or procurement is ordinarily made during the early stages of production or procurement on the basis of experience with similar products. In the absence of quality control procedures, the amount of inspection will, in general, be held constant throughout the history of production or procurement unless serious difficulties are encountered either in quality reactions or manpower shortages. In the case of tests and inspections on finished products or materials, the amount of inspection is often stipulated in an inspection procedure either referred to or included in the purchase specification, and exceptions or variations from the specified inspection are adopted only when practical considerations make some such modification almost imperative and then only after mutual consent of all concerned has been obtained.

One of the important developments in sampling and quality control work is the application of procedures whereby the amount of inspection called for depends upon the quality history of the product and hence may change from time to time as the quality history varies. These procedures are usually called "reduced inspection plans" since they usually permit, under specified conditions, a reduction in the amount of inspection below that called for in the standard inspection procedure. However, any sound reduced inspection plan must stipulate the conditions under which a return to normal inspection is to be made and therefore is really a plan for either decreasing or increasing the amount of inspection as the need for such a change is indicated by the inspection results. In fact there are instances where the principle is applied to establish procedures wherein the amount of inspection is increased beyond that called for in the standard procedure whenever information from prior lots indicates an unsatisfactory quality condition. This, of course, is done when an analysis of the situation reveals that the prescribed amount of inspection is not adequate under some of the conditions actually being experienced.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Bell Telephone Labs., Inc., New York, N. Y.

The concept of changing the amount of inspection when experience indicates the need for such a change is, of course, not new. For example, if the installation of a new machine results in the elimination of a certain type of defect which had occurred intermittently with the old machine, one may quite naturally reduce the amount of, or completely eliminate, inspection for this particular defect. Or again it may be found that raw material from a new source is quite variable and unpredictable with respect to a certain quality characteristic and one may increase the frequency with which checks are made on this particular raw material. These are simple illustrations of the basic principle upon which are based all statistical procedures for making the amount of inspection a function of the degree to which quality is controlled. This basic principle, expressed in statistical language, is that the risk of accepting unsatisfactory material through use of a sampling plan depends not only the particular sampling plan being used but also upon the likelihood that unsatisfactory material will be produced. Everyone engaged in setting up sampling plans uses this concept either consciously or unconsciously in a qualitative way all the time. The object of statistical procedures is to apply these principles on a quantitative basis and as a routine procedure so as to bring out immediately any need for a change in the amount of inspection.

TECHNIQUES

One of the earliest formalized sampling procedures wherein the amount of inspection is made to be a function of the control of quality is that of the Dodge-Romig Sampling Tables[2] which have been in use by industry since the late 1920's. These tables are designed for use in the inspection of lots of product consisting of a number of discrete articles and where the inspection is to be made on an attributes or go-no-go basis. Sampling tables of this type have been used more and more throughout industry during recent years and it will be helpful to study the important features of these sampling tables from the standpoint of the subject of this paper. This study will illustrate, by a highly successful application, the principles under discussion. Techniques for applying the same general principles to tests and inspections which perhaps are more generally of interest in A.S.T.M. work will then be discussed.

The Dodge-Romig sampling tables give sample sizes and acceptance criteria to be used in the inspection of lots consisting of discrete articles to determine which lots may be accepted without further inspection. It is assumed that each article inspected in the sample can be classified as either satisfactory or unsatisfactory (defective), and the tables give the number of defective articles which may be permitted in the sample from any lot without requiring that the remainder of the lot be inspected in detail. The sample sizes and acceptance numbers are listed as a function of the lot size and quality level which it is desired that the sampling plan enforce. In addition, the sample sizes and corresponding acceptance numbers are given as a function of the average quality of the product coming to the inspector, that is, as a function of the "process average." This is illustrated by Table I which is an extract from one of the Dodge-Romig tables.

---

[2] Harold F. Dodge and Harry G. Romig, "Sampling Inspection Tables," John Wiley and Sons, New York, N. Y. (1944).

From Table I it is seen that if one is inspecting a product in accordance with this particular sampling table, the sample sizes called for range from 36 to 300 depending upon what the process average of prior lots has been found to be. It is interesting to inquire how such sample sizes were arrived at. First it should be noted that this particular table is characterized by an AOQL (average outgoing quality limit) of 1.0 per cent. This means that any of the sampling plans given here will enforce (assuming 100 per cent inspection of rejected lots) an average outgoing quality of 1 per cent defective or better, and, in this sense, all of the sampling plans provide equal protection against unsatisfactory quality. Why then is the prescribed plan "Inspect 36, allow no defectives when process average is very good" but "inspect 300, allow 5 defectives when process average is relatively poor?" This came about purely from a consideration of minimizing inspection costs. It is a fundamental part of the inspection procedure of these AOQL tables that rejected lots be inspected 100 per cent to remove all defective articles. Therefore, the total inspection required for a series of lots consists of two parts, the sampling inspection which is made on every lot and the detailed 100 per cent inspection which is made on any rejected lots. The sample sizes given in the table have been calculated to be the particular ones which will result in the minimum amount of total inspection (sampling plus detailed) for the particular process average involved. Thus, it is seen that in this type of inspection a consideration of minimizing total inspection effort requires that the amount of sampling inspection be made a function of the degree to which quality is controlled. And in this case it was possible to obtain a mathematical solution which indicated exactly what the sample size should be in order to minimize total inspection costs.

TABLE I.—EXTRACT FROM DODGE-ROMIG SAMPLING TABLES (AOQL[a] = 1.0 PER CENT).

Lot Size	Process Average, per cent defective											
	0–0.02		0.03–0.20		0.21–0.40		0.41–0.60		0.61–0.80		0.81–1.00	
	n	*c*	*n*	*c*	*n*	*c*	*n*	*c*	*n*	*c*	*n*	*c*
4001–5000	36	0	85	1	135	2	190	3	245	4	300	5

n = sample size, *c* = allowable number of defectives.

[a] AOQL (average outgoing quality limit) is the poorest average quality possible in the accepted product if all lots rejected are inspected 100 per cent and accepted after removal of all defective articles.

The sampling procedure of this example is, of course, not applicable to many of the tests and inspections with which the A.S.T.M. is concerned. Many test procedures standardized by the A.S.T.M. involve a small number of tests (from 2 to 10) on each lot or batch of product, and quite often the test is one in which actual measurements are made on a variables basis. Furthermore, the product may be a bulk material such as a heat of steel or a bin of cement rather than a large number of discrete articles, and lots of such products cannot be made perfect by a detailed inspection to remove all defective parts. In the case of tests and inspections of this type, procedures for making adjustments in the amount of inspection are usually called "reduced inspection plans," and the establishment of suitable "eligibility" requirements together with the decision as to what is a safe reduction in inspection are the important steps in setting up such a plan for a particular job. Mathematical rules involving considerations of sampling risks, etc., do not lead to any unique solution in this case as in the case illustrated by the Dodge-Romig tables. However, such considerations do point out what the general nature of such requirements should be and give some indication of a quantitative nature in regard to the stringency of the eligibility requirements. Working on this basis a number of reduced inspection plans have been developed[3, 4, 5, 6, 7] and applied to a wide variety of products. Experience in these applications has shown that although many details of a satisfactory plan must be more or less tailor made for the particular application, there are certain general features common to most plans, and the techniques themselves may be classified into several more or less distinct categories. Table II presents the more basic features of four types of plans.

TABLE II.—FOUR TYPES OF REDUCED INSPECTION PLANS.

Plan	Eligibility Requirements	Reduction in Inspection
Type A	1. No lot failures[a] in last *k* (15–25) lots.	⅓ to ⅕ normal sample size
Type B	1. No lot failures in last *k* (10–20) lots. 2. Average fraction defective in last *k* (10–20) samples, less than allowable standard.	⅓ to ⅕ normal sample size
Type C	1. No lot failures in last *k* (10–20) lots. 2. Average of all measurements made in last *k* (10–20) lots better than standard value adopted.	⅓ to ⅕ normal sample size
Type D	1. No lot failures in last *k* (8–12) lots. 2. Average of all measurements made in last *k* (8–12) lots better than standard value adopted. 3. All sample averages and sample ranges for last *k* (8–12) lots exhibit satistical control.	⅕ to ⅛ normal sample size

[a] A "lot failure" is a failure to meet the acceptance criteria for a sample representing a lot or other unit of production. For example, if not more than one failure to meet requirements were allowed in a sample of five tests, a "lot failure" would occur if the sample representing a lot were found to have two or more failures to meet specification requirements.

Type A plan of Table II is, of course, very simple and involves little or no mathematical considerations. Such a plan simply states that if, lot after lot, no failures are encountered in a product from a particular source a reduction in the amount of inspection may be made. The virtue of such a plan lies primarily in its simplicity and ease of administration. However, with this extremely simple type of eligibility requirements, reductions in inspection are justified only after a considerable number of lots have been inspected. Such a plan is perhaps most useful in reducing the amount of inspection on certain quality characteristics while maintaining the normal amount of inspection on others. For example, in the case of chemical composition or impurities in materials it often happens that there are as many as 5 to 10 chemical components each of which must be maintained within specified limits. However, production from a particular supplier or from a particular source (quarry, mill, or furnace, for example) may never run into trouble with one or two of these components but may intermittently have trouble meeting the requirement for others. The particular component with which trouble is experienced may, of course, vary from time to time and it is only from a study of inspection results obtained on successive lots or batches that up-to-date conclusions can be drawn as to which components need the more frequent inspection. Under these conditions the simple rules of type A plan can be followed to deter-

---

[3] Harold F. Dodge, "Acceptance-Rejection Requirements in Specifications," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 877 (1934).

[4] Control Chart Method of Controlling Quality During Production, American War Standard Z1.3, p. 28, Am. Standards Assn. (1942).

[5] "Sampling Inspection," Statistical Research Group, Columbia University, McGraw Hill Book Co., Inc., New York, N. Y.

[6] G. R. Gause, "Quality Through Inspection, "*Army Ordnance*, Vol. XXIV, pp. 117–120 (1943).

[7] "A Sampling Procedure for Design Tests of Electron Tubes," *Industrial Quality Control*, Vol. III, No. 3. p. 19.

mine where a saving can be made in the number of chemical determinations.

Type B is quite similar to type A and likewise is notable primarily for its simplicity. Here there has been added a requirement that the average fraction defective in samples from the last *k* lots must not be greater than some adopted standard for allowable fraction defective. Therefore, type B is applicable rather than type A in those cases where a small percentage of test failures is to be expected even in perfectly satisfactory product. Such is often the case with "artificial tests" such as corrosion tests, simulated weathering tests, accelerated aging, or crazing tests which are either tests of "increased severity" or of somewhat indefinite relation to actual service condition. In this kind of test, the results are of an attribute nature and one cannot therefore use the more powerful techniques employing variables data as in plans of types C or D. On the other hand, this kind of test is usually relatively expensive and time-consuming, and it is particularly desirable to effect economies in inspection wherever quality history justifies it.

Type C is quite similar to type B but is for use where the inspection under consideration is of the variables type. In this case the grand average of all measurements in the last *k* samples is computed and compared with an adopted standard value. This standard value should of course be a somewhat tighter limit than the specification limit since specification limits, in general, apply to individual measurements or to averages of small samples. For example, if the characteristic under consideration were compressive strength of concrete cylinders and the specification limit were 3000 psi. for individual cylinders, one might adopt a standard value of 4000 psi. for averages of *k* samples for the purpose of determining eligibility for reduced inspection.

In none of the plans discussed so far is there a requirement that the inspection data indicate a state of "statistical control." Rather the requirements are, in essence, that recent inspection data indicate that successive lots consistently meet specification requirements. Plans of type D, however, incorporate the additional feature of requiring that there be evidence of statistical control. This involves the plotting of sample averages and sample ranges (maximum spread in sample) and the computation and plotting of control limits to make up the well-known control chart.[8] The work involved in these operations has been so systematized and simplified that only the simplest addition, subtraction, and multiplication are required. However, this added analysis of the inspection data opens a completely new field of vision into the quality behavior of the product which is being inspected. For example, if the inspection data do exhibit a state of statistical control it can be concluded that all of the variation in averages of samples selected from different lots is due either to quality variations within each lot or to errors of measurement. Therefore, the best estimate as to the true average for the last lot inspected is the average of all tests from the prior lots which exhibit a state of control. This estimate is, of course, much more reliable than is the average of the sample from the last lot, which average is ordinarily taken as the best estimate for that lot. Under this condition of statistical control a considerable reduction in inspection is justified.

Table II gives, in parentheses, numbers to indicate the usual range of the value of k applicable to each of the four different types of plans as well as the amount of reduction in inspection which is commonly employed. As indicated earlier, there is no mathematical solution which will determine what the exact values of any of these numbers should be as was the case with the Dodge-Romig tables. Rather the appropriate k value and the appropriate change in inspection is arrived at in each application through a consideration of the engineering, statistical, and practical aspects of the particular job. However, the values given in this table do represent values which have been found satisfactory in a number of applications. They are given here to indicate the order of magnitude involved and more particularly the relative order of magnitude for the four different plans. Plans of type A, while being simple and making use of what is perhaps the simplest of calculations, require data from a relatively large number of lots and justify a smaller reduction in inspection. Plans of type D, at the other extreme, involve more considerations and employ one of the most effective tools of modern quality control. They therefore require data from relatively few lots and at the same time justify larger reductions in inspection.

---

[8] See Supplement B of A.S.T.M. Manual on Presentation of Data. (Issued as separate publication, *STP No. 15-B.*)

The make-up of a reduced inspection plan need not, of course, be limited to the features illustrated in Table II. Individual plans for particular jobs may have many variations on or additions to the provisions of the four types of plans discussed here. However, many reduced inspection plans are similar to one of these four types, with special provisions possibly added because of engineering considerations peculiar to particular application. The conditions under which the different types are applicable are to some extent obvious from the nature of the plans themselves as described in the preceding paragraphs. Type D, since it employs the more powerful statistical methods is, of course, preferable from a purely theoretical standpoint. However, the use of this type of plan requires not only that inspection be by variables, but also that the measurements obtained be summarized continually and promptly. Furthermore, the plan must be applied to each quality characteristic separately. In those cases where a large number of quality characteristics are involved and the cost of inspection for each of them is relatively small it may be more economical to confine the inspection to one by attributes and employ a plan of type A or B. Even in those cases where the inspection results are of a variables nature it may, because of the paper work involved, be preferable to treat the data as attributes data (per cent failing to meet specification limits) and apply a plan of type A or B. One important advantage in treating inspection data on an attributes basis is the fact that results from a number of tests can be pooled together and only one statistical measure of quality performance used, namely, per cent failing to meet any of the tests or inspections involved. Quite often practical consideration (type of personnel available, time elements, availability of space, etc.) may more or less dictate what the character of a reduced inspection plan should be. Perhaps the only general rule to keep in mind is that more and more use is made of the inspection data as the type of plan used progresses from type A through type D. The more important the inspection characteristic under consideration and the more expensive the test, the more the effort which should be made to use a plan of type D. Plans of this type not only afford the greatest opportunity for savings in inspection cost, but the control chart record of inspection results yields valuable by-products. For example, the control chart is particularly helpful in indicating when and where to look for causes of trouble in the production process. By knowing when something has happened to affect quality adversely, steps can be taken to correct and remove the cause before material actually failing to meet specification requirements is produced.

One of the most important factors which determines the type of reduced inspection plan that should be adopted in any particular instance is the make-up of the inspection organization which is to carry out the inspection. Since the amount of inspection may change from time to time, the inspection organization and personnel available must be such as to make the changes practicable. The first reduced inspection plan introduced by any inspection organization may necessarily have to be rather simple and be designed to require only small and infrequent changes in the amount of inspection. As experience is gained in such applications and the benefits resulting are realized, it is often possible to rearrange the inspection organization or administrative procedures so as to permit the use of plans more sensitive to changes in quality levels. The whole problem of arranging the inspection organization and administrative procedures so as to permit the shifting of inspection effort from one job to another is a subject in itself and cannot be dealt with adequately in this paper. The only point to be made here is that the design of a reduced inspection plan and the make-up of the inspection organization must dovetail together, with changes possibly being made in one or the other from time to time in order to effect the greatest overall efficiency.

Table II indicates a reduction in the amount of inspection through a reduction in the size of the sample inspected from each lot or batch of product. However, in some instances the size of sample normally inspected is of the order of 1, 2, or 3 and little or no reduction can be made by this method. The economic urgency for reduction in inspection may not be too acute in these instances, and consequently there may be little need for application of these methods unless it be to establish a procedure for increasing the amount of inspection in order to obtain better discrimination between satisfactory and unsatisfactory lots. In this case the techniques used could be identical except that the specified amount of inspection would now be the "reduced inspection," and failure to qualify for this reduced inspection would call for a change to sample sizes larger than those specified. However, even in the case where only one test per lot or batch is specified, it sometimes becomes necessary (because of increased production, for example) to make some reduction in the amount of inspection per unit volume of production. In cases such as these the same techniques are sometimes applied except that reduced inspection involves "skipping" lots so that a test is made under reduced inspection on say, only one lot out of every three or every five lots. This procedure may seem somewhat drastic to some, but it must be remembered that the normal inspection itself, since it involves only one test per lot, quite likely affords, in itself, very little protection against passing limited quantities of product of inferior quality. In such cases the evidence of consistency from lot to lot afforded by the eligibility requirements of a reduced inspection plan may afford much greater assurance that the next lot produced will be satisfactory than will the evidence of a single test made on that lot. Furthermore, the situation may be that a reduction in inspection must be made in some way, the only question being, "Will the reduction be made across the board to apply to material from all sources (all suppliers, all production lines or all furnaces for example) or will an attempt be made to make the reduction on products from those sources which are well controlled, thereby distributing available inspection personnel and equipment on a more rational basis?"

Nothing has been included in Table II to indicate what criterion is to be used for determining acceptability of individual lots while on reduced inspection. This is a very important question, and fortunately a reasonable and practical solution is usually available. The procedure is usually to consider each lot inspected under reduced inspection as acceptable so long as the eligibility requirements continue to be met by the data up to and including that from the lot in question. However, as soon as eligibility is lost (for example, by a sample average being outside control limits) not only is the full sample size inspected in succeeding lots but a second sample is selected from the current lot, this second sample being of such a size as to bring the total sample up to the full normal sample size. Having then inspected the full sample from this lot, the usual acceptance criteria can be applied to determine acceptability of the lot. In this way rejection will be made only when a full sample as called for in the regular inspection practice has been inspected and found to fail the acceptance criteria. It should be noted that by this procedure it is possible to lose eligibility for reduced inspection without a lot actually being rejected. This, of course, is proper since reduced inspection is intended to be used only when quality is consistently better than the worst considered satisfactory.

SUMMARY

Efficient distribution of inspection effort requires that the amount of inspection be a function of the degree to which quality is controlled. Product from a source showing good control requires less inspection and testing than product from a source lacking such control. Simple summaries of inspection results obtained on samples from prior lots of product afford the necessary measures of the degree to which quality of that particular product is controlled and can be used to determine when the amount of inspection should be reduced or increased. Techniques for making these summaries range from a simple consideration of the number of failures in the last *k* lots tested to the relatively involved procedure of keeping a control chart record of inspection results. The simpler techniques involve little paper work but offer less possibility of large reduction in the amount of inspection. The more involved control chart method is indicated where the inspection is both highly important from a quality standpoint and also is costly to perform. Not only does this method offer the greatest savings in inspection cost but it also provides valuable auxiliary benefits such as assistance in controlling the production operations through the elimination of causes of trouble.

DISCUSSION

MR. O. P. BECKWITH.[1]—Will Mr. Gause please discuss the use of control charts employing limits for individuals.

MR. G. R. GAUSE (*author*).—Take for example a case where the normal amount of inspection is to make only one test from each heat of steel. In order to make any reduction in inspection we cannot cut down on the sample size. The method used in this particular case is called skipping lots; that is, a test was made on only every third heat rather than one test on each heat. Therefore, when we were on reduced inspection the average we plotted was the average of four successive tests. This, of course, was really the average over 12 successive heats, since we made only one test on every third heat.

In order to avoid remaining on reduced testing if something really went wrong, that is, in order to avoid remaining on reduced inspection until the entire 12 heats had been produced and we had thereby acquired the sample average to plot, we plotted the individual value as soon as it was obtained. For that reason, on the control chart were added the control limits for individuals.

The control limits, of course, are simply limits which indicate how much variation should be allowed in whatever you are plotting. If you are plotting averages the statistical theory indicates that you should allow or permit less variation in the sample average than you would in the case of individuals. So the control limits for individuals in this particular case were twice as wide, since the sample size was 4.

MR. ROBERT H. LACE.[2]—Referring to Table II on the fraction defective reduced sampling, if lots were continually accepted would not the fraction defective be much less than standard? In other words, if one had met item 1 one would automatically meet item 2.

MR. GAUSE.—Not necessarily. Of course, it will depend upon the standard and also upon the acceptance number. For example, as these plans are usually set up one might decide that 1 per cent defective is an acceptable quality level. The normal sampling plan might then consist of a sample of 100 from each lot, allowing three defectives in the sample of 100. If every lot were accepted, one could sometimes find 1, sometimes 2 and sometimes even 3 in the sample, without any rejections occurring.

However, the reduced inspection plan would probably require that the average fraction defective observed in samples from preceding lots should be less than ½ per cent to qualify for reduced inspection.

MR. LACE.—In other words, the standard for reduced inspection is frequently different and lower than that for acceptability?

MR. GAUSE.—Yes, let us say that we decide, for good engineering reasons, that 1 per cent defective product is satisfactory. It is not unusual in a lot of 1 per cent defective to get as many as three defectives in a sample of 100. Therefore, in order to avoid rejecting perfectly usable material we have to allow three in a sample of 100. So we say, as long as the process average is all right, we will allow three just to take care of sampling fluctuations. But before we will go to reduced inspection we will not only make sure that a large number of consecutive lots have met this requirement of not more than three defectives in a sample from any one of them, but we require also that the average in all samples must be less than probably something like $\frac{1}{2}$ per cent defective. You would not want to go on reduced inspection unless the process average is really somewhat better than the worst you would consider satisfactory.

---

[1] Chief, Product Engineering Lab., Alexander Smith and Sons Carpet Co., Yonkers, N. Y.

[2] Quality Control Manager, Ditto, Inc., Chicago, Ill.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON DEFORMATION OF METALS AS RELATED TO FORMING AND SERVICE

The Symposium on Deformation of Metals as Related to Forming and Service was presented at the Fifth Session of the Fifty-first Annual Meeting of the American Society for Testing Materials held in Detroit, Mich., on June 22, 1948. The symposium was sponsored by the Administrative Committee on Simulated Service Testing.

The last few years have witnessed a rapidly accelerating rate of accomplishment in the approaches to a more rational and fundamental concept of metal flow and fracture. The approaches vary from the more academic hypothesis and theories of the physical metallurgist and physicist to those of the works metallurgist in his laboratory and plant. The committee, therefore, felt it desirable to sponsor a symposium on this subject at this time. The following five papers were presented:

Tests of Ductility in Ship Structure—W. P. Roop
Notched-Sensitivity of Ship Plate—Correlation of Laboratory-Scale Tests with Large-Scale Plate Tests—N. A. Kahn and E. A. Imbembo
Measurement of Ductility in Sheet Metals—J. R. Low, Jr., and T. A. Prater
Hydraulic Bulge Testing of Sheet Metals—W. T. Lankford
Notch Bar Tension Tests on Annealed Carbon Steel Specimens of Various Sizes and Contours—M. L. Fried and G. Sachs

These papers together with discussion are issued as A.S.T.M. Special Technical Publication, No. 87, entitled, "Symposium on Deformation of Metals as Related to Forming and Service."

STATION DESIGN AND MATERIAL COMPOSITION AS FACTORS IN BOILER CORROSION

By R. B. Donworth[1]

Synopsis

The equipment in a modern power station is composed chiefly of two elements, iron and copper. Other elements, including zinc, chromium, molybdenum, nickel, mangenese, tungsten, cobalt, tin, phosphorus, antimony, arsenic, silver, and carbon, are also present in smaller quantities, and they are used principally for alloying purposes in order to obtain certain advantages not possible with simple elements. The purpose of this paper is to show the physical relationship of the materials and the influence of design on both corrosion and erosion and the subsequent carrying of the products into the boiler. The effects of such products in the boiler are the subject matter of other papers in this symposium.

Although James Watt would today scarcely recognize the power generating equipment which perpetuates his name, the steam part of the most up-to-date power station is in essence little different from that of his early successful design. The limits of pressure and vacuum have been increased, superheat has been added, and the turbine has replaced the reciprocating engine; but the use of partially expanded steam to heat the boiler feedwater is the only truly fundamental change in the cycle. Other changes, great as they are, represent development in degree rather than kind; they have not altered the basic simple process of burning fuel to generate steam which is transported through piping, expanded in a prime mover, condensed, and the condensate or equivalent returned to the boiler.

Improvements of vast importance have nevertheless been accomplished, and they are the result of tremendous striving for greater dependability and economy as well as lower investment costs. It may seem paradoxical that first cost can be reduced by the use of higher pressures and temperatures involving intricate boiler designs, complex controls, and many special aids to service and upkeep; but such is the case. This may be realized, however, by considering that an 850 psi. 900 F. station of today requires equipment to handle and burn only 65 per cent as much coal as a 200 psi. 500 F. station of the same capacity built 30 yr. ago, that the condenser is less than half as large, and that the throttle steam flow in terms of cubic feet per minute is only about 25 per cent as much. These reductions cut down the space requirement and the weight of steel and other materials needed both for equipment and building, and they more than offset the effect of increased

---

[1] Betterment Engineer, Power Stations Department, Duquesne Light Co., Pittsburgh, Pa.

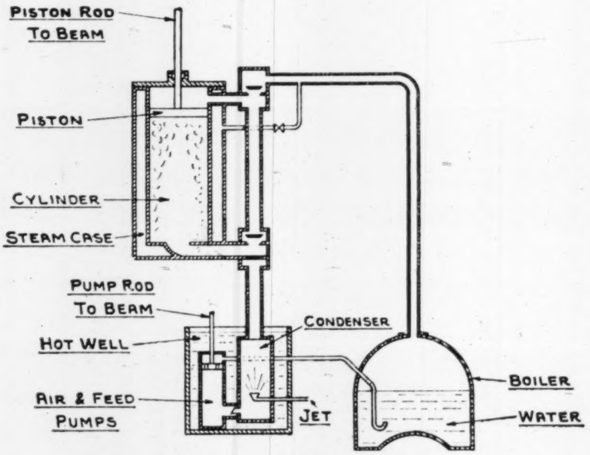


FIG. 1.—Diagram of Watt's Steam Cycle.

wall thicknesses and the use of special alloys required by the higher pressure and temperature. Comparative figures for a low- and a high-pressure station of these respective types are shown in Table I.

The development of higher pressures and temperatures has brought profound changes in the duties required of metals. Higher strength is needed to reduce the thickness and weight of pressure parts, and operation at elevated temperatures further accentuates this need. For these reasons carbon steel replaces cast iron as steam conditions approach 250 psi. and 500 F.; low-alloy steels become necessary at temperatures above 750 F., and the alloys become higher and more expensive as temperatures mount still further.[2]

Along with these developments it has become necessary to increase heat transfer rates, steam liberation rates, and other such factors, so that scale of any

TABLE I.—DATA COMPARING A MODERN POWER STATION WITH AN OLDER TYPE STATION.

	Station A	Station B	Values for B in per cent of A
Throttle steam pressure, psi.	200	850	
Total steam temperature, deg. Fahr.	500	900	
Station capacity, kw.	80 000	80 000	100
Number and size of units, kw.	2 at 40 000	1 at 80 000	
Type of compound	Cross	Tandem	
Number of boilers	20	2	
Fuel burned, tons per hr.	61.0	39.5	64.7
Steam flow, lb. per hr.	992 000	734 000	74.0
Steam flow, cu. ft. per min.	46 200	11 600	25.1
Number and size of steam leads, in.	2 at 24	1 at 14	
Weight of steam leads, lb. per ft.	2 at 171	1 at 131	38.3
Speed of H.P. element, rpm.	1 800	3 600	
Speed of L.P. element, rpm.	1 200	3 600	
Total floor area of turbo-generators, sq. ft.	3 550	1 540	43.4
Total weight of turbo-generators, tons	1 300	419	32.2
Condenser surface, sq. ft.	2 at 56 000	1 at 50 000	44.6
Area of boiler room, sq. ft.	16 700	10 032	60.1
Weight of boiler room equipment, tons	1 924	380	19.8

---

[2] The materials used are principally those specified by the ASME Boiler Code and Piping Code, published by the Am. Soc. of Mechanical Engrs. Comparable compositions are covered by A.S.T.M. Specifications A 106, A 158, A 206, A 217, etc., and B 12, B 13, B 111, etc.

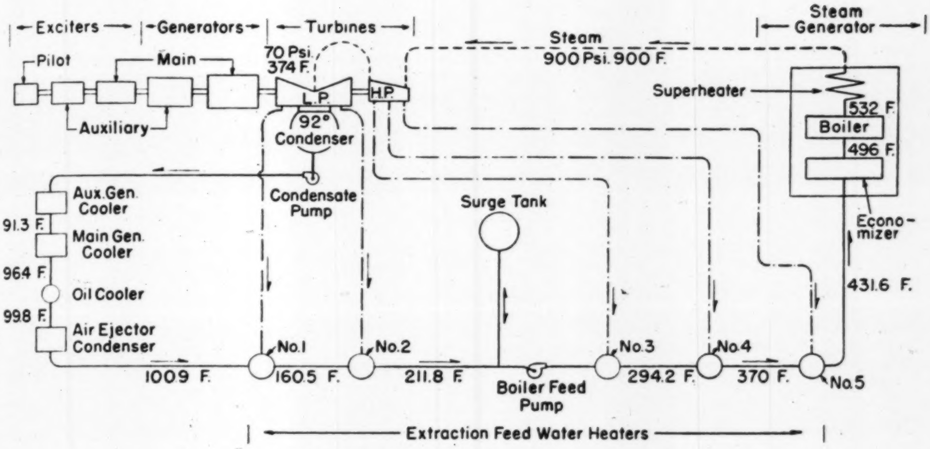

Exciters
Generators
Turbines
Steam Generator
Steam
900 Psi. 900 F.
Pilot
Main
Auxiliary
70 Psi.
374 F.
L.P.
H.P.
92°
Condenser
Superheater
532 F.
Boiler
496 F.
Econo-mizer
Condensate Pump
Aux. Gen. Cooler
91.3 F.
Main Gen. Cooler
96.4 F.
Oil Cooler
99.8 F.
Air Ejector Condenser
Surge Tank
431.6 F.
No.1
No.2
No.3
No.4
No.5
100.9 F.
160.5 F.
211.8 F.
Boiler Feed Pump
294.2 F.
370 F.
Extraction Feed Water Heaters

FIG. 2.—Simplified Diagram of a Regenerative Cycle.

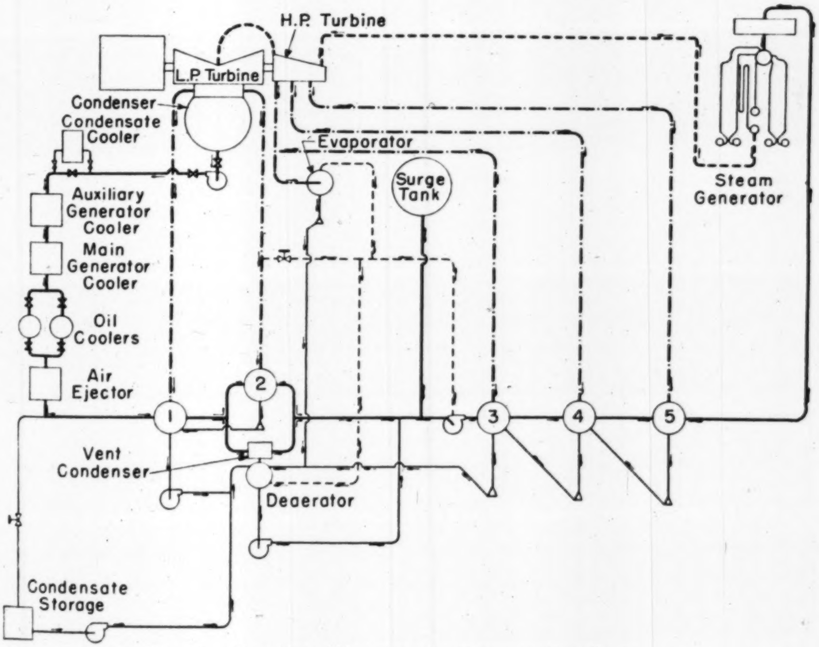

H.P. Turbine
L.P. Turbine
Condenser
Condensate Cooler
Evaporator
Surge Tank
Steam Generator
Auxiliary Generator Cooler
Main Generator Cooler
Oil Coolers
Air Ejector
1
2
3
4
5
Vent Condenser
Deaerator
Condensate Storage

FIG. 3.—Complete Diagram of a Regenerative Cycle.

appreciable thickness can no longer be tolerated on heat transfer surfaces. This has forced much improvement in the purity of water, which has in turn introduced corrosion problems that were entirely unknown in the earlier era. In addition greater reliability resulting in fewer and shorter outages is demanded, and this also contributes to lowered investment costs.

The development of the steam cycle is illustrated in Figs. 1, 2, and 3. In Fig. 1 is shown Watt's early cycle, and in Fig. 2 a simplified diagram of a present-day regenerative cycle. In the latter the cycle economy is improved through heating the feedwater to the optimum temperature with steam extracted from the turbine, the steam having performed useful work without the loss of heat to the condenser. The fuel consumption of the present-day power station is reduced approximately 7 per cent in comparison to a similar station employing auxiliary exhaust to heat feed water to 212 F.

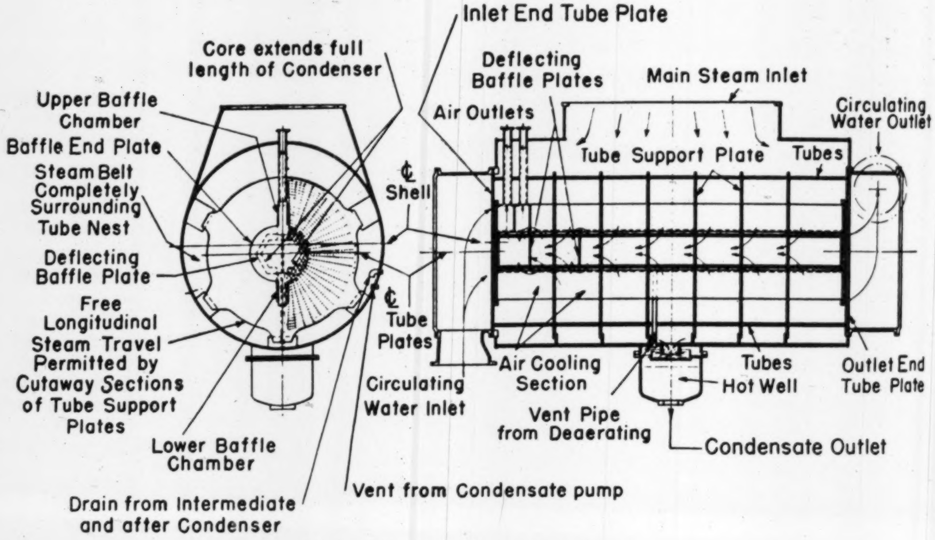

Inlet End Tube Plate
Core extends full length of Condenser
Deflecting Baffle Plates
Main Steam Inlet
Upper Baffle Chamber
Air Outlets
Circulating Water Outlet
Baffle End Plate
Tube Support Plate
Tubes
Steam Belt Completely Surrounding Tube Nest
℄ Shell
Deflecting Baffle Plate
Free Longitudinal Steam Travel Permitted by Cutaway Sections of Tube Support Plates
℄ Tube Plates
Air Cooling Section
Tubes
Outlet End Tube Plate
Circulating Water Inlet
Hot Well
Vent Pipe from Deaerating
Lower Baffle Chamber
Condensate Outlet
Vent from Condensate pump
Drain from Intermediate and after Condenser

FIG. 4.—Condenser Flow Diagram.

In Fig. 3 is shown a more complete diagram of the same modern regenerative cycle as that in Fig. 2. This diagram shows the addition of an evaporator, which takes steam from one extraction line and delivers vapor to the heater next below, and it also shows the cascading of drains from each heater to the next.

A TYPICAL CYCLE

For purposes of this paper, the fluid cycle is assumed to start at the boiler outlet. Under the most favorable conditions, the saturated steam contains some solid material carried over from the boiler, and it may also acquire materials from the attemperator which may be used to control final steam temperature. In the superheater this steam and its contaminants come in contact with alloy steels **(15)**[3] containing chromium, molybdenum, or perhaps nickel, and the piping and turbine parts contain the same materials in various proportions. Some of the substances in the steam deposit in the superheater and the turbine, and some pass through to reappear in the condensate. As the steam is expanded in the turbine, heat is converted into work, the temperature drops, the volume increases, and eventually the dew point is reached. After this point moisture forms in increasing quantity causing erosion of turbine blades and making it possible for some carry-over materials to redissolve. There have been instances of cracking of turbine blades located close to the dew point, and it is suggested here that this may be due to caustic embrittlement in cases where the boiler carry-over contains free caustic.

---

[3] The boldface numbers in parntheses refer to the list of references in the Combined Bibliography appended to these papers, see p. 940.

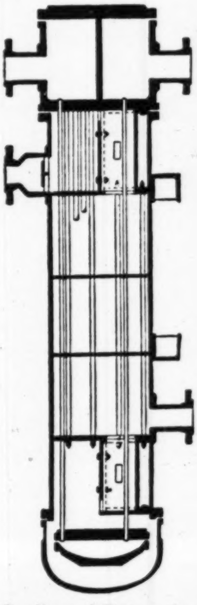


FIG. 5.—Outline of Extraction Heater.

The turbine exhaust containing approximately 10 per cent moisture then passes to the condenser at high velocity where as a hard driving rain it strikes tubes, baffles, shell, and other parts. The tubes form a very large area of nonferrous metal which is high in copper content and which may also contain zinc, nickel, tin, aluminum, phosphorus, antimony, arsenic, or any of a number of other materials.[2] The condenser shell and protective baffles are usually of steel or corrosion-resistant iron.

Thus the condensate leaving the condenser hot well may contain many metal contaminants as well as noncondensable gases and materials contained in the circulating water which may leak into the condensate space, all of this adding to materials carried over from the boiler water originally. The condensate is then moved by a hot well pump having cast iron and bronze parts through oil coolers, hydrogen coolers, ejector condensers, or other heat exchangers, all containing copper or admiralty metal tubes, and thence through one or more extraction heaters to the boiler feed pump where it is in contact with the chromium steel or chromium-nickel steel casing and a chromium-steel or monel rotor. It then passes through several more extraction heaters, the tubing of which is usually admiralty or an alloy of copper and nickel, and thence to the economizer and boiler where it is further heated to the boiling point, converted to steam, and superheated to the required temperature.

The piping up to the boiler is carbon steel, with cast iron valves on the low-pressure side of the boiler feed pump and steel valves on the high-pressure side, the former being commonly trimmed with bronze and the latter with chromium-nickel stainless steel.

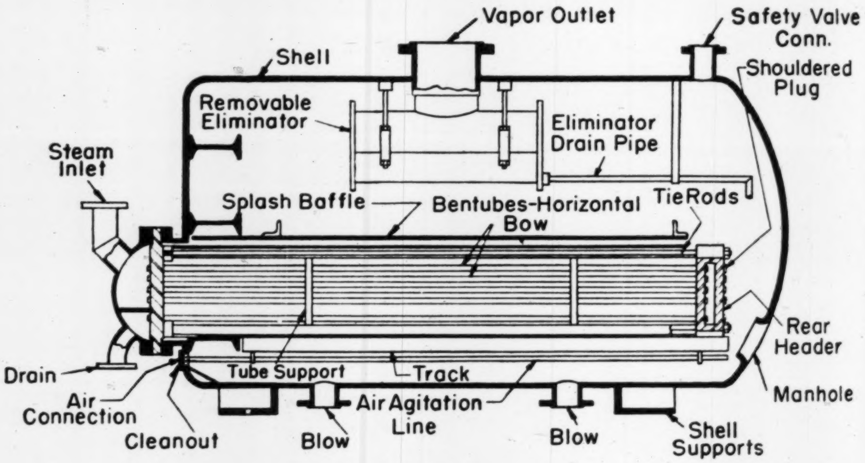

Vapor Outlet
Safety Valve Conn.
Shell
Shouldered Plug
Removable Eliminator
Eliminator Drain Pipe
Steam Inlet
Splash Baffle
Bentubes-Horizontal Bow
Tie Rods
Rear Header
Drain
Air Connection
Cleanout
Tube Support
Track
Air Agitation Line
Blow
Blow
Manhole
Shell Supports

FIG. 6.—Outline of Evaporator.

WATER THE GREAT SOLVENT

It is pertinent here to consider the compound known as water from a different viewpoint, namely, as a solvent, perhaps the greatest of all. It has been said (28) that "its action on nature's raw materials might well be compared to the prodigious appetites of swine and the promiscuous nibbling of billy goats; it absorbs large quantities of some materials and at least a small amount of everything with which it comes in contact." It is little wonder, therefore, that condensate, a relatively "pure" water, dissolves to some extent the various metals along its path through the entire system. This solvent action is tremendously enhanced by concurrent erosion, which may not only cut away the metal itself but may also remove protective products of corrosion, leaving fresh metal surfaces open to further attack (6, 7, 27).

EQUIPMENT AND MATERIALS

In Figs. 4, 5, and 6 are shown diagrams of several important pieces of equipment in which condensate makes contact with large areas of non-ferrous metal, namely the condenser, 50,000 sq. ft.; the extraction heaters, five of which total 24,500 sq. ft. including both inside and outside surfaces; and the evaporator, 340 sq. ft. These sizes refer to the requirements of the modern 80,000 kw. station referred to in Table I. In the condenser, steam strikes only the outside of the tubes (8), the inside being in contact with circulating water which is rejected from the plant. In the extraction heaters, however, the boiler feedwater commonly passes through the tubes while extracted steam is condensed on the outside and is returned through the heater drain piping, thus making both surfaces part of the feedwater system. In the evaporator, steam and drains touch the inside of the tubes and are returned to the feedwater system, while raw water to be evaporated makes contact with the outside.

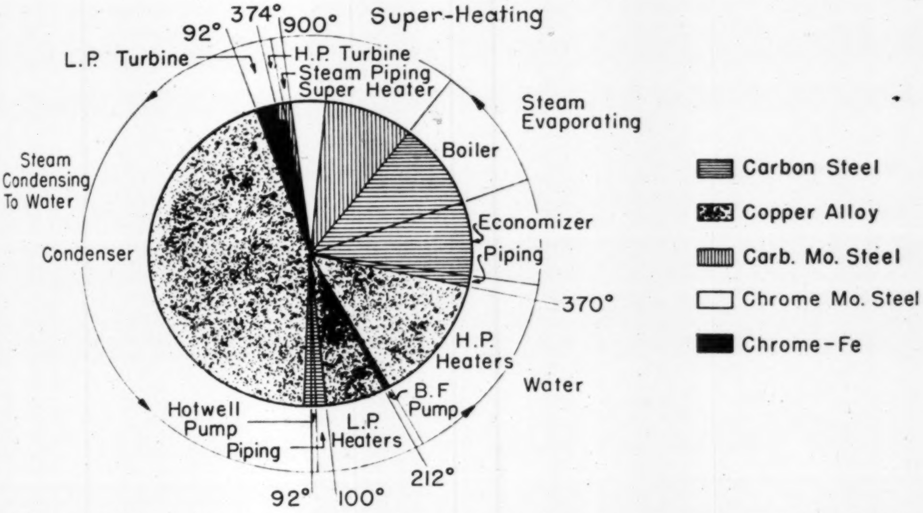


FIG. 7.—Relative Areas of Metals in Contact with Steam and Water in a Regenerative Cycle.

In Fig. 7 is a representation of the relative surface areas of different metals passed over by fluids employed in the cycle (steam and water). It is interesting to note that the total non-ferrous area substantially exceeds the ferrous area; and it will be readily realized that the fluids, in making a circuit through such a system, have ample opportunity to both corrode and erode these metals. Steam and water velocities in straight piping can be held to certain limits by the design, but very little control is possible in turbine nozzles and blades or in pump impellers and casings. Local high velocities due to eddying and swirling are difficult to prevent; and in water-steam mixtures the water particles seldom have the same velocity as the steam, and they do not follow the path which frequently must be designed for thermodynamic flow of the steam only.

INFLUENCE OF DESIGN ON CORROSION AND EROSION

In considering this whole problem of corrosion and erosion of metals outside the boiler, it is most important to realize that the power station design engineer today has very little choice of materials, since he must accept much which past experience has developed into current practice. Thus the great need for high-quality boiler water makes the surface condenser a necessity, and the complication of pumps and controls associated with contact type extraction heaters has definitely directed the trend to the surface type, both of which requirements at present dictate the use of tubes containing copper.

There are, however, many important situations in which much difficulty can be avoided by proper design. For example, ammonia corrosion of condenser tubes provides an easy way for copper to enter the feedwater in soluble form. Ammonia may enter the system with the raw water either through condenser leakage or with the evaporator vapor. An increase in the general corrosion of the steam side of the condenser tubes due to the presence of ammonia is naturally to be expected, but this in itself is not usually serious. However, condensed vapor dripping over an air offtake baffle may act as a fractionating column *locally* concentrating the ammonia sufficiently to remove copper in serious quantities. One solution of this problem is to provide equipment for removing the ammonia from the make-up water. A more practical solution in cases where the average quantity of ammonia is not too high, is to design the condenser properly with recognition of the manner in which ammonia concentrates. Corrosion by other dissolved gases such as carbon dioxide and hydrogen sulfide obviously must receive similar consideration.

Other instances where design has decreased or minimized the erosion-corrosion of metals include water separation and removal in the low-pressure end of steam turbines; protective baffles opposite the steam entrances to condensers and heaters; deaeration of condensate in the hot well or separate deaerator; arrangement of extraction heater drain equipment to reduce pipe erosion; and, perhaps greatest of all, the development of high initial steam temperatures and reheat temperatures to reduce turbine exhaust moisture.

It is apparent that, while the design engineer can do much toward reducing the erosion and corrosion of metals at many locations throughout the system, nevertheless, under the most favorable conditions some of the metals and their compounds will be transported to the boiler. Further improvement will therefore require much cooperative study and investigation on the part of the chemist, the metallurgist, and the engineer in order to discover and evaluate the effects of these foreign materials, to the end that the proper economic choices can be made for installations of the future. Discussion of some of these effects in the boiler will be left to other papers in this symposium, but it is strongly emphasized here that boiler design will no doubt play an important part in the ultimate solution of the problems.

DISCUSSION

Mr. R. C. Adams[1].—I should like Mr. Donworth to clarify his reasons for deaeration of drains only, instead of combining drains and condensate before the deaerator. His remarks implied that the dissolved-oxygen content of the deaerator effluent might be influenced by that of the deaerator influent. On the other hand, the condensate may not require deaeration so that a smaller unit will suffice for separate deaeration of drains.

The reason I ask is that extensive tests of deaerators which we have conducted, both shop and shipboard trials, have shown no relation between the degree of saturation of the deaerator influent and the completeness of deaeration of the effluent. Even with virtually saturated influent the deaerators discharged water with the same, low, dissolved-oxygen content as with good condensate feed.

Mr. R. B. Donworth (*author*).—Answering Mr. Adams question, I prefer to avoid large deaerators whenever it is possible, and to perform the deaeration in a properly designed condenser. However, since make-up, drips, and drains are usually very seriously contaminated, a small deaerator is usually required for them. From experience with both arrangements the condenser method appears to me to have three advantages:

1. I believe it to be cheaper in first cost.
2. It is far simpler and less subject to operating errors. Furthermore, the performance is less likely to be affected by inadequate maintenance.
3. The performance is more effective.

Suppose that the effluent from a good deaerating condenser amounts to 1,000,000 lb. per hour and contains 0.001 ppm. of dissolved oxygen; also that drips and make-up totaling 5000 lb. per hour containing 0.5 ppm. are to be added.

If these are mixed and passed through a large deaerator capable of producing 0.005 ppm. effluent, the entire flow of 1,005,000 lb. per hour will contain 0.005 ppm.

On the other hand, the drips and make-up may be passed through a small deaerator, which reduces the gas content to 0.005 ppm., and then mixed with the condensate containing 0.001 ppm. Calculations show that the dissolved oxygen content of the resulting mixture will be 0.00102 ppm., which is definitely better than 0.005 ppm. Comparative actual analyses may be inconclusive on this point, but one can nevertheless be sure that the second method produces purer water under the conditions assumed.

Mr. S. F. Whirl[2].—Mr. Adams' discussion of deaeration of boiler feedwater is very timely. Every power station design engineer is confronted with the problem of whether to provide a deaerator for all of the boiler feedwater or to rely on hot well deaeration with or without a small supplementary deaerator for make-up and drips and drains. A panel discussion on this controversial subject is scheduled for the Power Session of the Water Conference of the Engineers Society of Western Pennsylvania in Pittsburgh in October. This session will be co-sponsored by the Joint Research Committee on Boiler Feedwater Studies.

---

[1] Principal Chemical Engineer, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[2] Chief Chemist, Power Stations Dept., Duquesne Light Co., Pittsburgh, Pa.

MR. A. A. BERK[3].—I should like to comment on Mr. Donworth's theory attributing cracking of turbine blades to caustic embrittlement. It seems to me the theory can be verified very simply by temperature measurements. The concentrated caustic solutions, if they are there, would boil at a much higher temperature than water. In other words, if the dewpoint occurs at or near the expected temperature, there is no caustic present. If the dewpoint is found at a much higher temperature than expected, there is a strong possibility that caustic is there and may be a factor in the cracking.

MR. DONWORTH.—Mr. Berk's comment is very pertinent, but attention is called to the thermodynamics of steam flow through nozzles or the equivalent. As steam loses temperature due to such expansion, it does so because heat energy is converted into velocity energy, which is in turn given up to the turbine blades through transfer of momentum. I must confess I do not know very much about how that process takes place. The steam can lose pressure and temperature and acquire velocity with little increase in volume, although for the moment it still has all the energy it had at the start; but a part has been transformed from heat energy to velocity energy.

I do not know if under these conditions the actual dewpoint could appear ahead of the theoretical dewpoint. In other words, the steam just ahead of the blade row in which moisture first begins to appear may be sufficiently hot to fail to indicate moisture as suggested by Mr. Berk. It is also difficult to measure true temperatures under these conditions.

MR. D. S. MCKINNEY[4].—To add a little to this thought of Mr. Donworth's on the cracking of blades near the dewpoint, I want to point out that you do not have to have caustic to make metals of these alloys crack. I have done work on some stainless steels and in magnesium chloride solutions at 160 C. 18-8 stainless steel, under tension will crack in as little as 24 hr. So you may have a point although it may not be caustic that is causing the trouble. This points out another thing that Mr. Donworth mentioned. I believe many of these problems can be corrected by proper design. I do not know whether a turbine can be designed so that the blades are always under compression, but if you could I would predict that they would not crack.

MR. DONWORTH.—I know how it could be done. Suppose the turbine rotor was built as a hollow cylinder with the blades fastened at the outer end instead of the inner end. Centrifugal force would then put the blades under compression.

This might be done, but the problems would be tremendous, and other solution to the cracking difficulty will no doubt be sought first.

CHAIRMAN L. D. BETZ[5].—In making up the chart shown in Fig. 7 you considered the relative areas but did you consider the length of time that the steam was in contact with the various areas of different metals?

MR. DONWORTH.—The time element and also the velocity are no doubt very important in the processes of erosion, corrosion, and solution of the various metals. However, the chart was intended only as an indicator to assist in visualizing the situation, and it did not appear possible to include the time element and also retain the desired simplicity.

---

[3] Supervising Chemist, Boiler Water Research Section, U. S. Dept. of the Interior, Bureau of Mines, College Park, Md.

[4] Associate Professor, Carnegie Institute of Technology, Schenley Park, Pittsburgh, Pa.

[5] General Manager, W. H. & L. D. Betz, Philadelphia, Pa.

CORROSION OF HIGH-PRESSURE STEAM GENERATORS: STATUS OF OUR KNOWLEDGE OF THE EFFECT OF COPPER AND IRON OXIDE DEPOSITS IN STEAM GENERATING TUBES

By Richard C. Corey[1]

Synopsis

Numerous cases of severe, pit-type, internal corrosion in high-pressure steam generators, principally of furnace wall-tubes, have been ascribed to deposits consisting almost entirely of Fe_3O_4 and copper and its oxides. Opinions of the cause of this type of attack vary widely, but in the absence of conclusive experimental proof that thin deposits of Fe_3O_4, copper, or mixtures of these substances will cause pitting of steel under boiler conditions, the basis of the theories that have been proposed necessarily has been speculative. The present paper reviews critically published information on copper and iron oxide in boilers, and attempts to correlate it with the author's personal experiences with the problem and the meager amount of theoretical data that are available, in order to provide an objective background to the problem, and to evoke discussion from others who may have the evidence that is needed to establish definitely the effect of deposits of Fe_3O_4 and copper in boilers.

The question of whether or not deposits of copper and iron oxide have a specific effect in causing corrosion in high-pressure steam generators has interested boiler operators, manufacturers, and water-treatment specialists for several years. Particular attention was drawn to the matter during the period from 1941 to 1946 when a number of cases occurred of severe pitting and failure of furnace wall tubes in units operating up to 1400 psi., a characteristic of each case being that the pits were filled with dense plugs of Fe_3O_4 containing an appreciable amount of copper. Overheating was not a factor as the tubes were relatively free of calcium and magnesium deposits, and the microstructure in the fire side was normal. Consequently, there was considerable speculation as to whether the copper and the iron oxide were the *cause* or the *result* of attack, and diverse and conflicting theories, particularly with respect to the effect of copper, were proposed. The result has been that the matter is one of the most controversial in the technology of steam generation at high pressures.

The author is unaware of any conclusive experimental evidence, based upon experiments under controlled conditions of heat transfer; fluid temperature, velocity, and composition; and physical condition of the metal surface; that deposits of copper or iron oxide, or mixtures of these materials, have a specific effect in corroding steel tubes at nominal operating conditions of high pressure boilers. It is assumed, of course, that such deposits are not sufficiently thick to cause high-temperature corrosion, in the usual sense.

---

[1] Supervising Engineer, Combustion Research Section, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

Therefore, in lieu of direct experimental proof, for which it must be conceded there is no satisfactory substitute, a few facts known to the writer from his experience, and a review of the meager amount of relevant material that has been published will be presented in an effort to achieve two purposes: (*a*) To evoke extended discussion from others who may have the supplementary experiences and facts that are needed to place the question of the effect of copper and iron oxide in high-pressure units on a more substantial basis, and (*b*) to provide an objective background for fundamental research.

Copper and iron oxide each will be dissussed separately, since there is no reacon apparent at the present time why they should be discussed together. For each there will be given an annotated review of published material directly concerned with, and related to, their effect in boilers, followed by a quasi-statistical examination of theories, opinions, and pertinent facts, and a tentative program for fundamental research based thereon.

COPPER IN BOILERS

General:

It was about ten years ago that operators began to express concern over the possibility that copper-rich deposits in boilers might lead to internal corrosion. There were frequent reports of finding in drums, headers, and circulating tubes deposits that contained as much as 50 per cent copper, either as the metal or the oxide. Stimulated by speculation among some chemists that a couple might be formed between the copper and the steel with which it was in contact, the steel being anodic, there were recommendations that copper should be prevented from entering the feedwater cycle. This would have been a logical, but difficult, procedure had there been definite evidence that copper-rich deposits were directly the cause of the damage. There was no such evidence, however, at that time, and the whole subject was highly speculative. Opinions were sharply divided into two groups—one claiming without reservation that under normal operating conditions copper would cause localized attack of the tube metal with which it was in contact and the other claiming that in the absence of definite evidence from conclusive experiments in the field and in the laboratory, there was no cause for concern.

These diverse opinions became more apparent during a round-table discussion of boiler water problems held during the 1942 Annual Meeting of the Society.[2] There was extended discussion by the panel members and the audience of the effect of copper deposits in boilers, but for the very obvious reason that no one had definite evidence, pro or con, opinions were based on surmise, and the net result of the discussion was negative in so far as an answer to the problem was concerned. One purpose, however, was served by this panel discussion: it became clear that fundamental research and more conclusive evidence from actual tube failures were needed to establish, definitely, whether or not copper-rich deposits would cause corrosion attack of the boiler steel with which they were in contact.

Evidence of the confusion that has existed since copper first was mentioned as a possible source of corrosion in boilers may be found in the following annotated review of published material relating to the effect of copper in boilers.

Review:

Habib **(13)**[3] reported pit-type corrosion in the fire side of approximately 80 per cent of the generating tubes of a 750 psi. boiler and ascribed it to galvanic action between particles of copper deposited on the tube surfaces, the tube being anodic at the point of contact. One might justifiably suspect Habib's conclusion, however, on the basis that relatively large amounts of Fe_2O_3 and Fe_3O_4 were associated with the copper, and therefore it was possible that the copper was incidental to attack caused by other factors. In addition, no facts about water conditions were given, and the chemical equations presented to justify his theory of pitting would scarcely stand rigorous analysis.

---

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 977 (1942).

[3] The boldface numbers in parentheses refer to the list of references in the Combined Bibliography appended to these papers, see p. 940.

Owens (22) reported finding relatively large amounts of copper and CuO in deposits from a wide variety of component parts of the feed water and boiler water circuits of several medium- and high-pressure boilers. He concluded that "the presence of copper in some form in the deposits in boilers does not necessarily forecast corrosion. Hence, if this material is present, it need not cause undue apprehension. Its presence should, however, be sufficient to warrant intensive investigation and continual vigilance for any evidence of undesirable activity." Thus, although Owens did not dismiss the possibility that copper or copper-rich deposits would cause corrosion, it was implied that there was no direct evidence that copper, *per se*, would do so.

Several operating companies reported to the Edison Electric Institute (9) that copper was found in appreciable quantities where boiler-tube failures occurred, but only one ascribed the failures to the presence of copper. A later publication from the same organization, giving the consensus of 25 operating companies regarding the merits of the acid-cleaning of boilers, concluded that although boiler deposits consisted generally of Fe_3O_4 and copper, with occasional small amounts of calcium sulfate and silica, there did not appear to be any relation between the nature of the deposits and corrosion.

More informative than these routine reports from the Edison Electric Institute was a confidential report[4] by T. J. Finnegan for the Power Station Chemistry Subcommittee, Prime Movers Committee. Although this report was mainly for the purpose of summarizing the opinions of twenty-two member power companies and four boiler manufacturers regarding the effect of black iron oxide in boilers, interesting comments on copper also were presented. Finnegan wrote, "Copper in the boiler deposit is reported by many of the correspondents. While this is a separate problem and nothing has been demonstrated to show a definite connection with the problem of black oxide, the tone of several of the letters reveals a curosity as to whether or not some relation exists. As far as can be learned, however, the ordinary finely divided copper dispersed in the boiler sludge is harmless."

One correspondent reported finding several copper-colored balls, from $\frac{1}{4}$ to 1-in. in diameter, in the boiler during an outage. Examination of a cross-section revealed a core of Fe_3O_4 and finely divided copper, thin concentric laminations of hard, shiny copper and Fe_3O_4, and an outermost layer of metallic copper. It was believed the copper had deposited electrolytically on the Fe_3O_4, the laminations representing alternating periods when the copper concentration in the boiler was above or below some threshold value. The interesting hypothesis was proposed that suspended iron oxide in boilers may prevent the deposition of copper directly on the boiler metal, it being considered possible that pitting or grooving of the steel would result from galvanic action if copper plated directly on the steel.

---

[4] Permission to use this report was granted to the author through the courtesy of GeorgeDaniels, Chairman, Prime Movers Committee, Edison Electric Institute.

The fact in this case was that copper plated directly on the iron oxide, suggesting that Fe_3O_4 was anodic to copper under certain conditions in the boiler. Ordinarily, it might be expected that copper in boiler water is combined as an insoluble phosphate, oxide or complex compound, or as a stable complex hydroxy-anion, and therefore insufficient copper ion is available to oxidize the iron to Fe_3O_4. Furthermore, there is evidence that metallic copper is anodic to steel, as attested by occasional reports that copper plugs, gaskets and ferrules in contact with steel in economizers and drums corrode rapidly, particularly where the ratio of the exposed area of the copper to that of the steel with which it is in contact is relatively small.

Keeth (**19**) discussed copper plating of boiler drums and tubes and concluded that "Any corrosion which takes place as a result of the deposition of copper on boiler steel is assumed to be electrochemical in nature. Only when the action becomes concentrated in definite spots, as contrasted with uniform distribution over the entire drum, does it become actually dangerous. As stated before, it is a moot question just how serious this form of attack can become. A great deal is yet to be learned about it." This is another case where the author suggests that copper may cause corrosion in boilers, but in the absence of conclusive evidence, reserves final decision.

An unpublished report[5] from the Hall Laboratories, dealing with internal corrosion of a high-pressure boiler, discussed the possible effect of relatively large amounts of metallic copper, which occurred especially in pitted areas of wall tubes, and came to the interesting conclusion that the copper was the result of corrosion rather than the cause of it, hydrogen from corrosion due to *other* causes having reduced copper oxide in deposits in the tubes to metallic copper. The author has seen evidence that appears to corroborate this viewpoint. It has been noted, for example, that where localized internal corrosion of furnace wall tubes has occurred, the metal around damaged areas has a reddish "bloom," which microscopic and microchemical examination reveals to be a superficial coating of metallic copper.

Brister and Romer (**3**) discussed corrosion problems in high-temperature and high-pressure boilers, and reported finding metallic copper and copper oxide in several cases where severe pitting of steaming tubes occurred beneath plugs of iron oxide. They concluded that "when the combination of ammonia, copper, and oxygen exists, then we have at least one additional factor involved, namely, a galvanic couple which creates an anodic condition beneath the barnacles resulting in the corrosion of the metal . . ." And further, "From the corrective steps which have alleviated this trouble, it would appear as if copper and oxygen were the principal agents involved in this type of metal damage." These authors propose that copper deposits in boilers will cause corrosion by local cell action between the steel and the copper if once a pit has been formed as the result of dissolved oxygen corrosion. This viewpoint introduces an inconsistency that may not have been intended by these authors, since if it is assumed that iron is anodic to copper under boiler conditions, then the local cell formed between the steel and the copper would have the same polarity regardless of the disposition of the copper, and the need for a primary pit, due to dissolved oxygen, is not apparent. In other words, a small patch of copper deposited directly on sound boiler steel conceivably would have the same effect as if there were an intermediate layer of Fe_3O_4. The cell elements would be the closed-circuit type in which the current is a function of the polarization characteristics of the steel and copper under the stated conditions. Is there ample proof, however, that under the conditions stated by the authors the steel is anodic? How would one explain the corrosion of copper gaskets in contact with steel, under boiler conditions? Several possible theories occur to the author that might account for the difference in behavior, based upon complex galvanic cells and concentration cells, but such speculation seems futile in the absence of any data on the behavior of iron-copper couples at temperatures between 500 and 600 F., in boiler water, a relatively complex electrolyte.

---

[5] Made available to the author through the courtesy of R. E. Hall, Director, Hall Laboratories, Inc.

Measurement of the copper concentration in the feedwater cycle of a forced-circulation boiler was made recently (2) to determine the principal source, since appreciable quantities of copper were found in deposits from various parts of the boiler. Although these authors found no direct evidence that copper caused corrosion, they reserved decision over its ultimate effect for lack of conclusive experimental work. At the time this work was done, the main concern over the presence of iron and copper compounds in boiler feedwater was the fact that they formed adherent sludges on the boiler surfaces. There might be some question whether the copper in the sludge contributed to the adherence, but, again, there are no data to establish this point.

Corrosion of furnace wall tubes in a 1450 psi. unit, characterized by deep pits, was concluded (20) to be due to dissolved oxygen and relatively low alkalinity of the boiler water, but the presence of considerable quantities of copper in the deposits in the damaged tubes suggested that ". . . copper might act as an accelerating agent for corrosion," and one of the steps taken to improve conditions was to reduce the ammonia entering the system. Thus, these authors attributed the initial attack of the tubes to causes other than copper but considered the possibility that copper may have a secondary effect, thereby following somewhat the same reasoning as that of Brister and Romer (3).

Straub reported (25) on severe pitting of wall tubes, associated with copper-rich deposits, in high-pressure boilers and concluded that despite theories to the contrary, there was no evidence that copper was the cause of corrosion in the cases he had observed. The author followed closely one of the cases that Straub described and came to the same conclusion. The boiler in question operated for about ten years with relatively few wall tube failures. Late in 1944 a wall tube ruptured, and subsequent examination of a number of other tubes showed severe, extensive pitting. During a period of about three months, 4 tubes were replaced because of leaks or ruptures, and 91 because of severe internal pitting. Although there was appreciable copper in the deposits of Fe_3O_4 associated with the damaged tubes, all the facts in the case suggested that the pitting was caused by oxygen, either during outages or during operation, and that the pits became foci of rapid attack during protracted periods of operation at full load, incurred by wartime demands. As will be shown later in the discussion of iron oxide in boilers, there is reason to believe that once a pit has started in a steaming tube, under certain conditions the base of the pits will corrode at an accelerated rate as it becomes deeper. The mechanism of this type of attack is probably very similar to that ascribed to corrosion in crevices.

For what it is worth, it may be mentioned that about eighteen months prior to the failures and the discovery of extensive pitting in one of the units described by Straub, the use of a proprietary oxygen scavenger, described as "colloidal" iron, was discontinued, since it was believed to have caused bulging of a few wall tubes by forming an excessively thick scale of Fe_3O_4. (But the unit operated for 6 yr. with this compound without damage to the tubes.) It might be reasoned that the suspended iron oxide produced by the compound prevented copper from plating on the metal, in accordance with Finnegan's[4] suggestion, and that when the use of the compound was discontinued, copper plated on the metal and established a galvanic cell, with the steel being the anode. This, however, would be specious reasoning in the present case, because other changes occurred in the boiler water during the time the iron scavenger was discontinued and the failures occurred, and it would be wrong to impute the damage to the tubes to this one factor.

Hankison and Baker discussed (**14**) wall tube damage similar to that described by Mitsch and Yeager (**20**) and Straub (**25**), stating that the severe pitting they found originated from causes other than copper, and concluded that "There is no positive evidence that copper enters into, or is a part of this type of corrosion, even though copper and/or copper oxides are frequently found in the barnacle strata." The barnacles to which the authors referred were hard, dense plugs of Fe_3O_4 in the pits.

The question of the effect of copper in boilers is subject of a current controversy in England. Parkhouse (**23**) believes that the copper occurs in the feedwater as copper bicarbonate, converts to copper hydroxide in the boiler as the result of reaction with the water-conditioning chemicals, and then becomes associated with calcium phosphate sludges. He attributes the corrosion of desuperheater tube bundles to the accumulation of a copper-rich sludge around the base of the bundles, causing electrolytic action between the copper and the iron. He states that the removal of ammonia from the feed water is not sufficient to prevent attack of the copper, and presents the results of experiments made at room temperature and at 212 F., which purport to show that (*a*) NH_3-free feedwater will dissolve as much copper as water containing 2 ppm. ammonium bicarbonate, and (*b*) an excess of sulfite and a pH of 8.0, maintained with NaOH, are required to prevent the attack of copper. He concedes, however, that ". . . at 360 F. and 1000 lb. pressure a different story might result." Since Parkhouse's experiments all were made under non-flow conditions and at temperatures likely only to occur in surface condensers, deaerating heaters and low-pressure extraction heaters, he failed even to approximate the flow and temperature conditions in high-pressure stage heaters, in which the pickup of copper is very likely to be appreciable. His data are condensed in Table I.

TABLE I.—CORROSION OF COPPER TUBES IN WATER CONTAINING AMMONIA.
(Specimens in contact with solution for one week, at room temperature)

NH_3, ppm.	NaOH, ppm.	Na_2SO_3, ppm.	Copper in Solution in One Week, ppm.
0.0	0.0	0.0	0.05
2.0	0.0	0.0	1.00
2.0	1.0	0.0	0.75
2.0	10.0	0.0	0.05
2.0	0.0	excess	0.50
2.0	0.6	excess	nil

It is observed that the copper concentration was negligible when sulfite was maintained in excess. Whether this was due to the removal of the dissolved oxygen or to some specific effect on the copper of the sulfite ion is not clear. A study should be made with similar solutions that have been deaerated mechanically.

It is indeed unfortunate that Parkhouse did not investigate more thoroughly the cause of corrosion of the desuperheater tubes, rather than assuming it was caused by galvanic action between steel tubes and the copper in the associated deposits.

Gillham and Howes took strong exception (11) to Parkhouse's conclusions, stating that in their experience corrosion of bronze and copper components in feedwater with a pH of approximately 8.5 and containing less than 0.01 ppm. of dissolved oxygen has shown that "... the corrosive effect of ammonia in the feedwater is very definitely enhanced by increased temperature and pressure." They state also that "... corrosion was experienced resulting in generator tube failures due to copper deposits."

Gaunt (10) does not agree with Gillham and Howe that the corrosion of generating tubes is due to copper deposits and states that "Although deposits high in copper have been found generally throughout the boilers with which I am concerned, *no corrosion has resulted from its presence.* X-ray powder photographs have shown that the copper in these particular deposits is present both as oxide and in its elementary form." He concurs with Partridge and Hall that copper is an incidental product in the corrosion process. In the plant to which Gaunt refers, the treatment initially was to add sulfite, hydroxide and phosphate to the feed pump suction, the dissolved oxygen in the feedwater being consistently less than 0.01 ppm. and the NH_3 about 0.05 ppm. Later, boiler water was re-circulated to the economizer inlet and sulfite only was added to the feed pump suction. He reports that no noticeable difference in the amount of copper in the boiler resulted from this change.

Conclusions:

The opinions expressed in the foregoing review may be divided into the three categories, as set forth in Table II.

Only limited conclusions can be drawn from a summary of this type, since it is based on limited information given in the various publications, and therefore the different categories cannot be weighted with respect to other factors that might be significant. It is apparent from context, however, that the assumption that active attack of boiler steel will result from contact with copper or copper-rich deposits is based upon extrapolation of the reversible potential of the Fe-Cu couple, measured under highly specific conditions of temperature and type of electrolyte, to the markedly dissimilar conditions of boiler operation. There are serious objections to extrapolations of this kind, particularly where all the variables and their relative effects are not known. In the present case, the effect on the Fe-Cu couple of saturation temperatures, fluid velocities, and the water compositions of high-pressure boilers is not known.

TABLE II.—SUMMARY OF OPINIONS ON THE EFFECT OF COPPER DEPOSITS IN BOILERS BASED UPON REVIEW OF LITERATURE SINCE 1938.

Basic Opinion	Favoring
1. Boiler steel, in contact with copper or copper-rich deposits at boiler operating conditions, will corrode as the result of galvanic action	5
2. Concede possibility that the action described above will occur, but reserve decision because of lack of experimental proof	5
3. No evidence that copper will cause corrosion and believe copper is the result of corrosion due to other causes	5

The reversible cell potential of a bimetal couple, which is measured isothermally and reversibly in a specific electrolyte, may be used to predict the *tendency* of a given metal to corrode under the conditions that the potential is measured, but frequently it is very difficult to identify the rate-controlling factors. Furthermore, it is not uncommon for the polarity of the elements of a bimetal couple to reverse when conditions are changed. Therefore, when the electrode processes of a given couple are not completely known, thermodynamic calculations to determine, *a priori*, the behavior of the couple are not justified, and experiments simulating as closely as possible the conditions under which the couple is used must be made.

Consider the reversible cell potentials of copper in water with and without ammonia:

E° 25 (volts)

$$2\,Cu + 2\,H_2O \rightarrow 2\,Cu^{+} + H_2 + 2\,OH^{-} \ldots\ldots -1.35 \ldots\ldots (1)$$

$$2\,Cu + 4\,NH_3 + 2\,H_2O \rightarrow 2\,Cu(NH_3)_2^{+} + H_2 + 2\,OH^{-} \ldots\ 0.11 \ldots\ldots (2)$$

$$Cu + 2\,H_2O \rightarrow Cu^{++} + H_2 + 2\,OH^{-} \ldots\ldots -1.175 \ldots (3)$$

$$Cu + 4\,NH_3 + 2\,H_2O \rightarrow Cu(NH_3)_4^{++} + H_2 + 2\,OH^{-} \ldots\ 0.05 \ldots\ldots (4)$$

Thus, the formation of cuprous and cupric ions is strongly favored by the presence of ammonia, as indicated by the large shift of the potential in the positive direction. Nevertheless, studies of the rate of solution of copper in water containing ammonia show that the rate is negligible unless dissolved oxygen is present, which appears to reduce the hydrogen overvoltage of the copper. This illustrates for one case the limited value of the reversible cell potential in estimating the rate that a metal will corrode and the necessity for knowing the complete system with regard to such factors as overvoltage and nature of the corrosion products.

It is pertinent to mention at this point that it is improbable that the primary reactions of copper in the feedwater system are as simple as shown in Eq. 1 to 4, since the condensate leaving the surface condensers usually has sufficient dissolved CO_2 to have an appreciable effect on the corrosion of preboiler equipment with copper and brass components. Tronstad and Veimo (26) found a marked increase in the solubility of copper in water at 65 F. when CO_2 was present and attributed it to the formation of complex copper compounds.

Some experiments made by Gould and Evans (12) suggest that care be used in deciding the effect of copper on steel. They considered the possibility that copper deposited as a discontinuous layer on steel might intensify attack of the steel as the result of the Fe-Cu couple. To test this, low-carbon steel specimens were plated with copper, engraved with a scratch line to expose a localized area of steel, where the current density would be extremely high, and then placed in a hot salt solution. Less action at the scratch line was observed than was anticipated, and at times copper redeposited on the scratch line and stifled attack. In order to secure a large exposed area of steel, the specimens were plated with copper over one-half their width and exposed to boiling distilled water for periods of 6 and 76 days. In one test, the water was maintained free of dissolved oxygen, and in the other the specimen was open to the air and was aerated 1 min. each day. Uncoppered specimens were tested simultaneously for comparison. The results are given in Table III.

TABLE III.—CORROSION OF PLAIN AND HALF-COPPERED STEEL SPECIMENS IN DISTILLED WATER AT ABOUT 212 F.

Specimen	Duration of Test, days	Weight Change After Pickling, mg.	
		Deaerated	Aerated
Uncoppered steel	6	−2.6	−23.2
	76	−0.5	−365.2
Half-coppered steel...	6	−20.1	−45.0
	76	−2.2	−342.0

It should be noted that although the half-coppered specimen corroded appreciably more in the 6-day test than the plain one, the amount of corrosion in the 76-day test was approximately the same for both specimens, under both conditions of aeration. It was reported that the copper had disappeared at the end of the 76-day tests. The authors are of the opinion that although copper is cathodic to iron in acid and neutral solutions, it may be anodic to iron in alkaline solutions, where iron tends to become passive and copper to dissolve with the formation of complex anions. They state, "... it is not impossible that a state (pH) may be reached where iron is incapable of depositing metallic copper, and where metallic copper already deposited will return to the solution." They suggest tentatively, until further investigations have been made, that copper compounds in boilers do not cause corrosion where "... water conditions are *normal*, but that they increase the potential danger that intense attack might arise if, locally and temporarily, water conditions should become *abnormal*." Assigning quantitative terms to "normal" and "abnormal" boiler water would be the principal purpose of any investigation of this problem, and it is obvious that the scope would necessarily be extremely broad to evaluate properly all of the variables.

The present status of the problem of copper in boilers is stated concisely in a recently published handbook on corrosion (17): "The significance of these copper deposits and their relation to corrosion of steel in boilers is a controversial subject among water-treating authorities. Some claim that copper is but an innocent bystander in the corrosion process; others hold it to be a serious offender in promoting pitting-type attack by acting as a cathode to the steel of the boiler. Neither viewpoint has been substantiated by direct experimental proof."

The proof required should be based in part upon controlled laboratory experiments simulating as closely as possible the conditions of high-pressure boiler operation with the objective of answering the following questions:

1. Is metallic copper anodic or cathodic to boiler steel, or does the polarity change with conditions such as temperature, relative distribution of the copper and composition of the boiler water?
2. What conditions are necessary for metallic copper to plate on the boiler surfaces?
3. In what forms other than metallic does copper occur in boiler sludges and deposits?
4. How is copper combined in the feedwater and the boiler water?
5. Does Fe_3O_4 react with metallic copper or the copper compounds in oxide sludges and deposits, and what is the nature of the reaction?
6. Does copper within a pit in the boiler steel accelerate attack in the pit, and is the mechanism different than when metallic copper is plated on a normal steel surface?

There are many aspects to each of these phases of the problem, and considerable care will be required in selecting the independent variables and the parameters; but until such a research program has been completed, the effect of copper in boilers will remain controversial.

IRON OXIDE IN BOILERS

General:

Concern over the possible effect of iron oxide deposits in boilers arose early during the late war when a number of unusual serious failures of wall tubes occurred in several medium- to high-pressure boilers, and caused unscheduled outages in utility and industrial plants during period of extreme emergency.

To attest the seriousness of these failures, the tube damage shown in Fig. 1 is typical, the furnace side of the tube having ruptured extensively and in a brittle manner, the edges of the fracture showing little or no elongation.

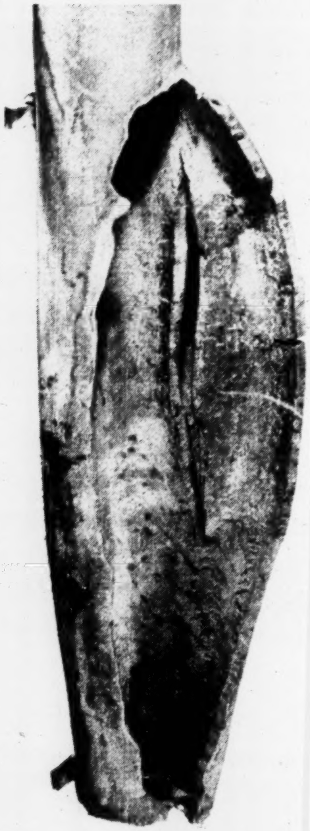


FIG. 1.—Ruptured Furnace Wall Tube from 1400 psi. Unit.

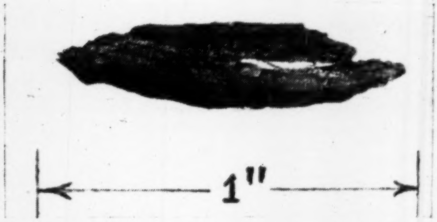


FIG. 3.—Plug of Iron Oxide from Pit in Water Wall Tube.

In the author's experience, each of these failures was characterized by small holes or extensive cracks, resulting from penetration of the tube wall by internal pits in the furnace side of the tubes. The pits ranged from small, localized ones to long, shallow ones, such as shown in Figure 2 and, as a significant characteristic, were filled with either a soft, spongy mass or a hard, dense, brittle scale. Very often the material could be removed from the pit as a solid plug, which had the contour of the pit where it had been in contact with the metal. Physically, all samples the author examined were black to reddish black, strongly magnetic, and in the case of the solid plugs, laminations and sharp cleavage planes were clearly evident in the cross-section. One of these is shown in Fig. 3. They were composed almost en-

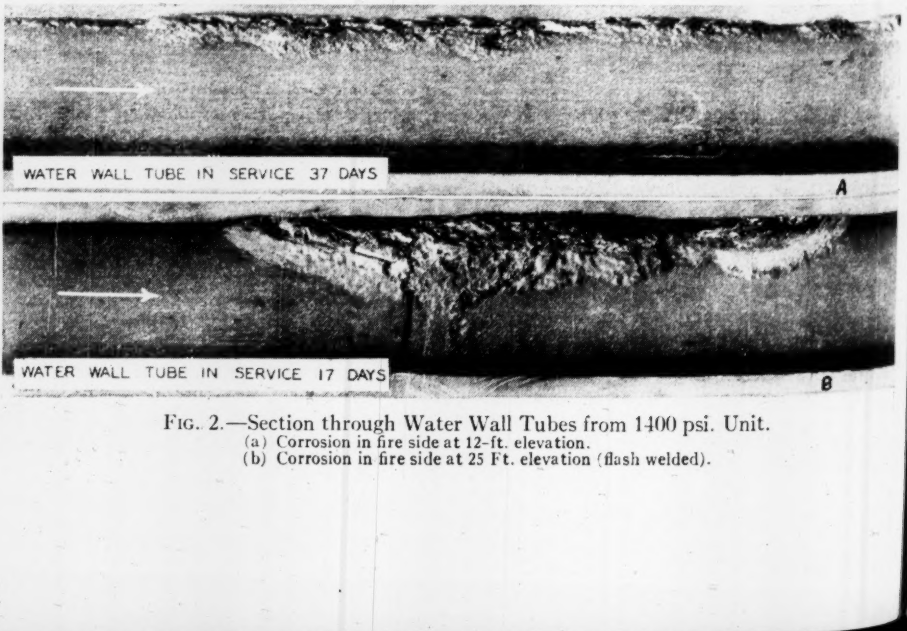

WATER WALL TUBE IN SERVICE 37 DAYS
A
WATER WALL TUBE IN SERVICE 17 DAYS
B

FIG. 2.—Section through Water Wall Tubes from 1400 psi. Unit.
(a) Corrosion in fire side at 12-ft. elevation.
(b) Corrosion in fire side at 25 Ft. elevation (flash welded).

tirely of magnetic iron oxide, Fe_3O_4, and copper as the metal or one of its oxides, the latter frequently comprising as much as 25 per cent of the total. Although Fe_3O_4 was the predominant substance associated with these failures, and therefore believed to have a dominant part in causing the corrosion attack, copper also was suspected, since it appeared not only to be distributed quite uniformly throughout the oxide in the pits, but also as an extremely thin film of copper or cuprous oxide, appearing as a reddish "bloom" on the metal, for a considerable area around the point where tubes failed.

At least seven different theories have been proposed to explain this type of corrosion, and, as in the case of copper lack of experimental proof makes it impossible at the present time to state beyond all doubt the effect of iron oxide. Possibly no single theory would be applicable to all cases that have been reported, due to the numerous factors unique to boiler operation that may cause pitting of tubes, but the problem can be simply resolved into two questions: (*a*) What causes the initial pit? and (*b*) what factors accelerate attack within a pit once it has formed? The latter question has an extremely important bearing on the problem, as there are several cases of wall tube corrosion on record in which pits were known to be present for several years, but which progressed very slowly until some change in conditions caused them to penetrate the tube wall within a relatively short time. Similarly, cases have been reported in which remedial steps have arrested tube failures, but pits antedating the failures are known yet to be present.

A review of published information on wall tube failures of this type will afford a background to the problem.

Review:

The Prime Movers Committee of the Edison Electric Institute published statements (9) from various operating companies covering approximately the period 1940 to 1944, in which a brief, general description was given of tube failures in plants of the respective companies. Although the information given was rather sketchy, it was evident that a few cases of wall tube damage similar to that already described had been experienced and that the type of deposit found where corrosion occurred was probably the same. Opinions of the cause of the attack varied widely, and no clear-cut conclusion could be drawn. Information given in this publication that is pertinent to this paper will be summarized later.

The report by T. J. Finnegan of the Power Station Chemistry Subcommittee, Prime Movers Committee, Edison Electric Institute, referred to previously,[4] presented information on iron oxide in boilers submitted by representatives of twenty-two power companies and four boiler manufacturers. Based upon the concensus of the majority, the following significant conclusion was offered. Iron oxide usually is found in boilers in two forms; one, a loose powdery sludge, considered to originate from preboiler equipment and believed to be harmless unless allowed to accumulate to the extent that overheating of heat absorbing surfaces occurs; the other, a hard, dense continuous scale that is usually found where pitting, bulging or cracking of tubes has occurred and generally believed not to be the cause but the result of corrosion from other factors.

One correspondent reported an unusual observation that is difficult to explain. Regularly, after starting up a boiler, the boiler water contained appreciable amounts of suspended Fe_3O_4 for a few days, which was not considered with alarm. On two occasions, however, suspended oxide appeared in the water after the unit had been in operation for some time, and tube failures occurred within a week. The author recalls a similar phenomenon, which at the time was attributed to hypercritical observations by the plant chemist but in retrospect may have some basis in fact. He noticed that THQ, used for routine sulfate determinations, underwent peculiar color changes approximately 24 hr. before wall tubes failed. An unusual amount of iron oxide and hydrogen, probable precursors of tube failures resulting from overheating, may have caused the color changes noted.

It was not until the 1946 Annual Meeting of The American Society of Mechanical Engineers that a complete, candid discussion was given of several instances where wall tube damage of this type occurred. Experiences at the Springdale station were discussed by Hankison and Baker (**14**); the Firestone, Mystic and Fisk, stations by Straub (**25**); and the West End station by Mitsch and Yeager (**20**).

Extended discussion of the corrosion at Firestone was published (**5**) a short time later to describe in detail the experiments and observations made over a long period of time by the boiler manufacturer to determine the fluid circulation and tube metal temperature characteristics of the water walls. As in most cases of this kind, defective circulation was one of the factors suspected, and although it is not possible yet to state unqualifiedly what the minimum fluid velocity should be in the wall circuits of a given unit to prevent attack, the following circumstances at Firestone appeared to rule the fluid velocity in the wall circuits from consideration: (1) The average velocity in the water walls was the same as in a number of other units of similar design in which no tube damage had occurred; (2) the failures of wall tubes due to internal corrosion did not occur until about six years after the unit was in operation, at which time they occurred suddenly and in considerable number; (3) with no subsequent change in practice except to modify somewhat the water-treatment schedule, and to acid-wash the unit, no wall tube failures have occurred subsequently during a period of over two years.

Brister and Romer (**3**) have discussed similar failures in high-pressure boilers, stating "In the areas which we are discussing, metallic copper, oxides of copper, and black oxides of iron are present in large quantities. These deposits may be soft or in the form of hard 'barnacles.' Underneath these deposits large corrosion pits form, and beneath these pits the metal undergoes serious deterioration in the form of intergranular disintegration."

All of the cases of wall tube corrosion mentioned above occurred in natural-circulation boilers operating at a gage pressure up to 1400 psi. Wall tube corrosion in a gage pressure at 2000 psi., forced-circulation boiler also has been reported (**2, 4, 24**) and since deposits rich in iron oxide were found where tube failures occurred, a special investigation (**2**) was made to determine the sources of the iorn oxides. The authors stated, "Over the past few years the occurrence of iron oxide in boilers has become a matter of interest . . . to operators of high-pressure boilers, as the presence of this substance must always indicate corrosion in some part of the system. Usually, the amount of oxide is small by comparison with the large area of steel surfaces involved, and the indicated loss of metal, if uniformly distributed, is not large enough to be a cause of great concern. If, however, there are small areas of attack, the amount of oxide may well indicate serious corrosion leading to failures." The deposits in question occurred in the furnace side of the tubes as a hard, adherent scale and on the casing side as a soft mud that dried out as a dust. The most unusual feature of some of the wall tubes was the pitting found beneath small nodules of oxide. It was found generally that regardless of the rate of circulation, the wall tubes all had a thin coating of black oxide, the furnace side being noticeably cleaner than the casing side, and that the pitting beneath the small oxide nodules was more pronounced on the casing side where no heat was received from the furnace than on the furnace side of the tubes. One of these tubes is shown in Fig. 4.

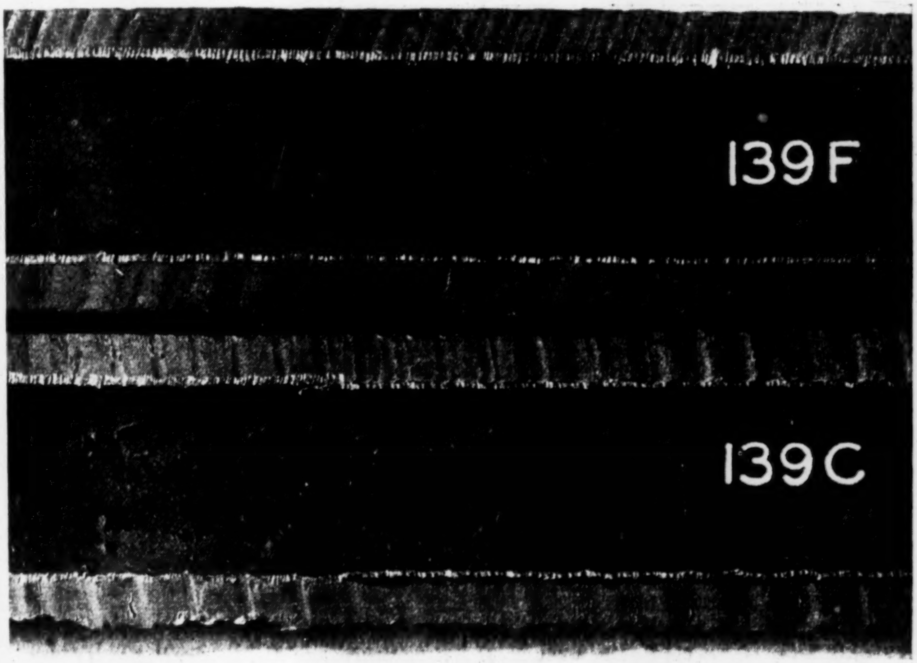


FIG. 4.—Pitting in Casing-Side of Tube from Forced-Circulation Unit.

Discussion and Conclusions:

Although there may be other important variables in the type of wall tube damage described above it is certain that the following factors are common to each case:

(*a*) The corrosion occurs in vertical tubes, and therefore probably is due to causes other than those ascribed to corrosion of low-pitched generating or floor tubes.

(*b*) The deposits associated with the corrosion consist principally of iron oxide and copper or its oxides, and little or none of the other varieties of scale.

(*c*) The failures, in the most serious cases, are brittle ones, with little or no evidence of plastic deformation of the tube walls. Bulging of tubes has occurred in numerous instances where the same type of scale was found as with brittle failures, and it is conceivable that the mechanism of corrosion attack prior to the deformation of the metal was the same; bulging being a secondary result of the initial attack.

It appeared possible, therefore, that careful study of certain factors in each case that has been reported, or in the author's experience, might provide the evidence needed to establish whether or

TABLE IV.—SUMMARY OF AVAILABLE DATA ON WALL TUBE FAILURES IN BOILERS OPERATING AT A GAGE PRESSURE BETWEEN 500–2000 PSI.

	Case I	Case II	Case III	Case IV	Case V	Case VI	Case VII*	Case VIII	Case IX	Case X
Type of internal corrosion[a]	1A, C	1A	1A	1A, C	1A	1B	1A, B	1B	1A	1A
Location of corrosion[b]	2A, C	2C	2A	2A	2A	2B	2B	2D	2A	2A
General condition of tubes[c]	3A, C	3B	3A	3A	...	3C	3C	3A***	...	...
Type of failure[d]	4A, B	4C	4C	4A, B, C	4C	4B, C	4A, B, C	4C	...	...
Microstructure adjacent to pits[e]	5A	5A	5B	5A	...	5A	5A	...	...	...
Boiler pressure, gage pressure, psi	1400	1325	1250	1350	1450	500	...	1900	1350	1350
Feedwater:										
Type of makeup	evap.	evap.	evap.	evap.	evap.	evap.	...	evap.	...	...
Dissolved O_2**	yes	yes	no	yes	yes	yes	yes	no	...	...
Ammonia	yes	...	...	yes	yes	yes	yes	yes	...	...
Boiler water:										
Analysis[f]	8A	8B	8A	8A	8B	8E	...	8A	...	...
pH	9.0–10.0	10.5	11.5	11.1	...	10.0	10.0–11.5	10.5	...	...
Acid washing before or after trouble	after	before	after	before	...	after	...	before	before	before
Operation before failure occurred, years	10	3 1/2	1	7	3	8	...	3	1 1/4	...
Suggested causes of corrosion[g]	11A, B, D, E	11B, C	11C	11A, B, F	11B, E, G	11A, B, D	11B, F, G	11H	11H	11H
Remedial measures[h]	12A, B, C, D, E	12C, F	12D	12C	12B, C	12A, C, E	12B, C	12G	...	...

[a] 1A—Pits filled with dense brittle scale consisting mainly of Fe_3O_4 and copper.
1B—Pits filled with soft deposit consisting mainly of Fe_3O_4 and copper.
1C—Same as 1A or 1B and dense plugs of Fe_3O_4 found lying loose in lower headers and floor tubes prior to failures.

[b] 2A—Furnace-side of tubes, mainly in zone of high heat absorption.
2B—Furnace-side of tubes, over entire furnace wall.
2C—Furnace-side of tubes adjacent to circumferential field or shop welds.
2D—Casing-side of tubes as well as in furnace-side.

[c] 3A—Clean except for thin deposit of Fe_3O_4 throughout tube.
3B—Phosphate or silicate scale associated with main deposit or scale.
3C—Appreciable deposit of Cu or Cu_2O adjacent to damaged areas.

[d] 4A—Extensive fracture with little or no deformation of tube wall.
4B—Bulging of tube prior to failure.
4C—Small pinholes or cracks.

[e] 5A—Intergranular attack extending appreciable distance into metal.
5B—Normal structure as compared with undamaged metal in same tube.

[f] 8A—Phosphate, excess OH, oxygen scavenger
8B—Phosphate, excess OH, no oxygen scavenger
8C—Coordinated phosphate, oxygen scavenger
8D—Coordinated phosphate, no oxygen scavenger
8E—No treatment used.
Refers to treatment in effect a reasonable time before failure occurred.

[g] 11A—Pitting started during boiler outages through contact of wet surfaces with air or by refilling boiler with undeaerated condensate of relatively low pH.
11B—Excessive dissolved oxygen feedwater, liberated at steaming surfaces and caused pitting.
11C—Fe from preboiler equipment produced sludge on tube in which free OH concentrated and caused accelerated attack.
11D—Concentrated film-boiling due to inadequate circulation.
11E—Boiler water pH lower than generally considered safe.
11F—Acid-cleaning destroyed normal protective film on metal and small oxide nodules formed as in 11A.
11G—Copper deposits caused attack by local cell action, steel anodic.
11H—Scale or sludge caused overheating.

[h] 12A—Filled boiler after outage with alkaline, deaerated water.
12B—Increased pH of boiler water.
12C—Removed all O_2 with excess scavenger.
12D—Carried lower "B" reading, or used coordinated phosphate control.
12E—Acid-washed unit to remove all scale and deposits.
12F—Reduced SiO_2 in feed to minimum.
12G—Reduced Fe and Cu entering boiler to a minimum.

* General survey of several high-pressure boilers.
** Dissolved oxygen in amounts 0.05 to 0.3 ppm. reported.
*** Furnace side cleaner than casing-side of tubes

not the deposits are the *cause* of corrosion or the *result* of it. Accordingly, Table IV was prepared to show as many as possible of the significant factors associated with ten recorded cases of wall tube corrosion in medium- to high-pressure boilers.

It will be noted that in general the

boiler pressure, type of make-up, and location and kind of damage to the tubes, are similar. With one exception, case VI, conventional methods of water treatment were used, minor variations being reflected in the amount of excess hydroxyl ion and pH of the boiler water. Evidence of intergranular attack in damaged areas was found in five of six cases reporting, and where deep-etch tests were made, the metal was characteristically discolored in the region of grain boundary damage. Here, however, the similarity of the cases ends.

Frequent failures occurred between one and ten years after the units were placed in operation; the majority giving trouble after at least three years of service.

In some cases the failures preceded acid washing, and followed in others, there being no obvious relationship between corrosion of wall tubes and acid washing. For example, in case I no failures occurred in over two years since acid washing, but in case IV no trouble was experienced until after the units had been acid washed. Further to obscure the possible effects of acid washing is the fact that after cleaning the boilers in this manner other steps were taken simultaneously to arrest attack, so it is impossible to isolate the effect of any single factor.

The causes ascribed to the pitting and failure of tubes vary widely. Generally, more than one theory has been proposed for a given case, depending largely on the number of responsible individuals involved. In a few instances, however, it was believed that no single theory could account for the corrosion, so a sequence of events was proposed. For example, dissolved oxygen may have initiated pitting according to 11A or 11B, and subsequent concentration of hydroxyl ion in the oxide filling the pit accelerated attack within.

The remedial measures, too, varied widely, and as in so many problems of this type, expediency made it impossible to adjust one variable at a time; consequently, the important factor or factors are not discernible. Consider cases III, IV, and VIII, in which only one step was taken to arrest attack, and it is seen that each was different; a lower "B" reading or coordinated phosphate control was used, all dissolved oxygen was removed, and iron and copper entering the boiler was reduced to a minimum.

It is possibly significant that in six of the eight cases for which information on the remedial measures was available, dissolved oxygen was reduced to a minimum. This, however, is accepted good practice, and because of lack of an established value for the amount of dissolved oxygen that can be tolerated in high-pressure boilers, or of any data showing the percentage of the feedwater oxygen occurring in the downcomers, it is usual to reduce the oxygen in the feedwater to less than 0.05 ppm.

Frequently, intergranular attack of grain boundaries in metal adjacent to pits is suggested as evidence that fluid velocities in wall circuits are too low. The author, however, contends that this is not unequivocal evidence of faulty circulation, since nothing is known of the events within a pit in a steaming tube. For example, in one case intergranular attack was found in the metal at the base of an oxide-filled pit, at a point where no heat was received from the furnace. Furthermore, measurements showed the fluid velocity in the wall circuits to be adequate. In another case, although a deep-etch specimen from a pitted area indicated severe intergranular attack as shown in Fig. 5, the pH, free hydroxyl ion and total dissolved solids all were relatively low, as no water treatment was used. In both of these cases the units were operated over a period of eight to

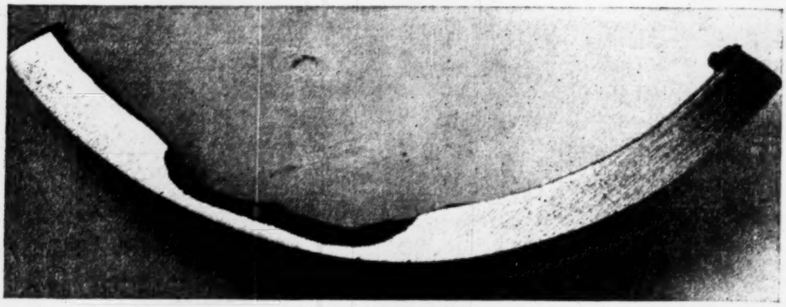


FIG. 5.—Macroetched Wall-Tube Specimen, 500 psi. Unit.
(Etched in hot 20 per cent HCL.)

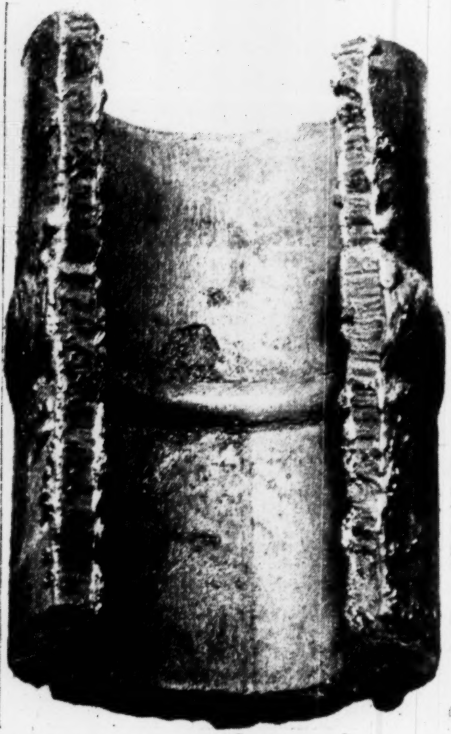


FIG. 6.—Flash-Weld in High Pressure Wall Tube.
(In service about two weeks.)

ten years with relatively little trouble, but during the war, when sustained full, and frequently excessive, steam loads were required, failures occurred suddenly and frequently. It seemed illogical to attribute the difficulties to deficient circulation rates under these conditions.

Research urgently is needed to determine the factors that accelerate corrosion within a pit, once it has formed. A satisfactory explanation thus may be obtained for the fact that several boilers operated for a number of years without unusual trouble, particularly where the usual types of scale were absent, and then with no significant change in operating conditions, frequent wall tube failures occurred.

In case I, wall tubes were badly pitted, and as one corrective step the unit was acid-cleaned for the purpose of removing scale from existing pits. Apparently this procedure was effective. In case IV, however, oxide-filled pits are known still to be present, but reduction of dissolved oxygen entering the boiler to 0.02 ppm. has stopped tube failures. How can these apparent contradictions be reconciled?

A peculiar condition noted at the time when frequent wall tube failures were occurring in case I was rapid corrosion adjacent to flash welds in wall tubes, located 24 ft. above the furnace floor. A typical example of this kind of corrosion is shown in Fig. 2(*b*), the tube having been in service approximately two years. Another flash-welded tube from the same furnace, shown in Fig. 6, was in service for about two weeks, and it is obvious that the rate of attack of metal adjacent to the weld had been very rapid. To ascribe the attack as due simply to a hydraulic disturbance near the slight projection of the weld does not account wholly for this condition, since severe attack has been found for a considerable distance upstream as well as downstream from the weld. The metallurgical characteristics of welds are inherently different from the tube steel, and the two might conceivably establish an electrochemical couple in which the maximum current density would occur in the transition structure, which is where attack appears to start. It should be considered, therefore, that the composition, temperature and velocity of the fluid passing such a welded area has a marked effect on the behavior of the cell, and changes in any of the variables might result in accelerated attack. It has been suggested that boiler water pH values of the order of 9.0 to 9.5 lead to corrosion in such welds. Although there is some precedent for this, nothing definite is known.

It must be concluded that although some of the worst cases of corrosion of wall tubes appear to have been corrected, neither the cause nor the prevention of attack are clearly defined. Conclusions derived from the quasi-statistical examination of the available data are limited by lack of knowledge of the specific effect of the numerous factors that appear to be involved and by the fact that in no case were the variables under sufficient control to permit their evaluation. Despite these shortcomings, there are recurrent factors in the ten cases cited from which tentative conclusions may be drawn as a basis for fundamental research and as a guide in arresting corrosion of wall tubes where internal scale is not a factor.

1. Effect of Iron Oxide.—There are indications that iron oxide has no specific part in the corrosion of furnace wall tubes; that is, any other material distributed in the same manner on the boiler surfaces, either as a compact scale in a pit or as a soft sludge throughout the tube, would have the same effect. There is merit in the contention that iron oxide from preboiler equipment and suspended in the water is harmless unless it bakes on the tubes as a hard sludge and causes overheating. Also to be considered is the possibility that cracks in such a deposit, in which boiler water may concentrate, will cause localized corrosion under conditions that are as yet undefined. The hard, dense Fe_3O_4 found in pits is probably a result of corrosion and may have a part in the corrosion mechanism within the pit, but there is no evidence that it functions other than as a semipermeable barrier between the boiler water and the metal comprising the pit; another substance of the same physical characteristics might have the same effect.

2. Effect of Pits.—Under conditions as yet undefined, pits in furnace wall tubes are potential foci of accelerated attack. The oxide with which they are filled is essentially a semi-permeable matrix in which boiler water salts can concentrate and lead to attack in a manner similar to that which occurs in crevices (**29**): that is, by direct chemical attack and by rather complex concentration cell processes. Further, the presence of a discontinuity in the surface of a steaming tube, such as is presented by an oxide-filled pit, might conceivably cause a type of nucleate boiling, where the specific rate of release of steam bubbles would be high and therefore have the effect of intensifying attack at such a point. The dynamics are obscure of steam release from surfaces in high-pressure boilers, as affected by fluid pressure, density, viscosity, slip velocity and surface tension, and by the tube diameter, thickness and quality of the surface.

3. Cause of Pits.—The procedures for draining and refilling a boiler have an important part in causing pitting of boiler surfaces. Poor practice in these respects can lead to extensive pitting, which may go unnoticed until attack within the pits is accelerated by unusual demands on the boiler or by radical changes in the water treatment schedule. It is more than coincidence that the frequency of wall tube failures increased during the past war. The over-all appearance of numerous tubes examined by the author suggested that the oxide-filled pits were of long standing, probably originating during outages or after refilling the boiler with poorly deaerated water of too low a pH.

Dissolved oxygen in the feedwater also may account for pitting of steaming surfaces during operation, and good practice requires that the concentration in the feed be maintained as low as possible, preferably less than 0.05 ppm. Unfortunately, however, tolerances have never been established, and this, considered with the fact that the ratio of the oxygen in the feed to that in the downcomers is very rarely known for any given unit, suggests an important problem in field and laboratory research. Too frequently, all the corrosion ills of boilers are attributed to dissolved oxygen, with the result that in many cases the real cause is overlooked.

4. *Fluid Velocity in Wall Tubes.*—The fact that four of the worst cases of wall tube corrosion on record were corrected by means other than increasing the circulation rate suggests that although this factor must be taken into account, it is the least likely one in cases where the boiler has operated for several years without unusual wall tube losses.

A Research Program:

Any fundamental studies to establish the effect of iron oxide in high-pressure boilers, with special reference to wall-tube corrosion, should consider the following factors:

(*a*) The conditions favoring accelerated attack within oxide-filled pits. The effect of boiler water temperature, composition, and pH and the rate of heat transfer through the tube should be determined. This is a program of large scope but of great importance to present-day practice and possibly to trends to higher steam pressure and temperatures and rates of heat transfer.

(*b*) The maximum dissolved oxygen that can be tolerated in a steaming circuit without causing pit-type corrosion.

(*c*) The effect of uniform deposits of iron oxide or other insoluble phases on steaming surfaces, in so far as they may lead to intense corrosion at discontinuities in the coating.

Since considerable iron oxide originates from uniform corrosion of preboiler equipment, there is definite need for data on the rate of corrosion of low-carbon steel by oxygen-free condensate, and for fundamental studies of the properties of ferrous hydroxide, with special reference to the reaction:

$$3\ Fe(OH)_2 \rightarrow Fe_3O_4 + 2\ H_2O + H_2$$

For example, determinations should be made of the rate of corrosion of boiler steel in water nominally of feedwater composition as a function of temperature, velocity, and pH. The data from such studies would establish the optimum pH for protection of preboiler and boiler surfaces in contact with evaporated makeup and condensate. Complementing this work, time-potential data should be obtained under the same conditions to establish the polarization characteristics of the steel.

The chemistry of ferrous hydroxide is not completely understood, and since it is considered always as the primary product in the corrosion of iron, information is needed on its solubility and pH in pure oxygen-free water, and its rate of decomposition, according to the reaction above, as a function of temperature and pH.

Thus, our knowledge of the effect of copper and iron oxide deposits in boilers is meager and unsatisfactory and is based largely upon speculation. Each is a separate problem, meriting intensive study to establish facts important not only to boiler practice today but to future developments in steam generation. The path to these facts consists of objective data of patient, objective research and not of unmeasured speculation.

DISCUSSION

Mr. S. F. Whirl.[1]—This paper very effectively summarizes the various viewpoints and interpretations that have been placed upon the occurrence of copper within boilers and their influence on corrosion. Judiciously, the author has emphasized that the information he presents is entirely speculative. He is to be commended not only for a well written paper, but for his analysis of the problem.

There are many cases on record where no apparent corrosion has been encountered in the presence of deposits rich in copper; in contrast, there have been cases of rather severe corrosion where copper appeared to play a part. Could this difference in behavior be attributed to or associated with the presence or absence of other elements such as zinc and nickel? Could the state of subdivision of the copper be a factor? It is common knowledge that copper alloy losses in equipment by erosion are frequently just as great or greater than those due to simple corrosion or solution. Would the metal eroded from copper alloys and carried into the boiler as a suspension behave the same as that dissolved in the feedwater?

I agree with the author that this problem requires fundamental research for its solution. In actual practice it is most difficult and often impossible to isolate the specific cause or causes of a failure since the chemical, design, and operational phases of the problem are so closely interrelated.

Mr. Frank E. Clarke.[2]—Experience at this station agrees with the author's conclusion that there is little evidence to support the fear that copper-bearing deposits cause boiler corrosion. The water-sides of Naval boilers normally are covered with thin sludge films which contain metal oxide corrosion products and phosphate reaction products of the disodium phosphate treatment. It is not unusual for these deposits to contain high concentrations of copper and zinc compounds (10 to 30 per cent of each). Free copper particles also are common. These non-ferrous sludges do not appear to cause corrosion of the tube metal. Like other sludges, they are objectionable in that they tend to bake on the watersides and to cause heat blistering if inadequate blowdown is applied.

In a few instances, copper-plating and peculiar localized waterside corrosion have occurred simultaneously in Navy boiler tubes. In these instances, corrosion existed as localized clusters of extremely deep hair-line fissures and pinholes. The uncorroded, cathode areas which separated the pits were heavily plated with bright metallic copper. The uncorroded portions of the tube contained normal copper-bearing sludge films of the type described above. Concentration of this damage in high heat transfer sections of water-walled tubes suggested that the metal loss resulted from stress corrosion and that copper-plating was caused by the consequent abundant discharge of electrons in the cathode areas. There was no reason to suspect that copper initiated the attack.

---

[1] Chief Chemist, Power Stations Dept. Duquesne Light Co., Pittsburgh, Pa.

[2] Chemical Lab., U. S. Naval Engineering Experiment Station, Annapolis, Md.

A comprehensive research program such as that recommended by the author is the only practicable means of resolving the many mysteries and fears now associated with non-ferrous boiler deposits.

MR. F. N. ALQUIST[3] (*presented in written form*).—During the past ten years our organization has been obtaining data both on copper and iron oxide in boilers. The purpose of this discussion is to add to the information contained in the paper data on X-ray diffraction analyses of 15 boiler deposits containing copper. The second purpose is an attempt to obtain a quantitative idea of the amount of iron oxides found in operating boilers.

The data (**1**) in the accompanying Table V show the X-ray diffraction analysis of 15 deposits from boilers containing copper. The copper varies from 5 to 25 per cent.

The problem of the iron oxides and the amounts found in boilers is of primary importance. Uhlig's (**30**)[3a] new book on corrosion states, "Fundamentally, from a standpoint of corrosion, a boiler is but a thin film of magnetic iron oxide supported by steel. This oxide film is continually being damaged and repaired during boiler operation, with corresponding production hydrogen. Since the surface film of magnetic iron oxide is the major protection of the steel, it must be maintained in such condition that it is least permeable to water."

The accompanying Table VI is based on field studies of 14 boilers. These boilers, after varying periods of operation, were chemically cleaned using inhibited hydrochloric acid. Quantitative analysis of the drain acids for iron and acid together with volumes of acid and surface areas provided the information for the calculations. In this table the operational corrosion found in these boilers in cases 7 through 20 indicate that 1.00 to 5.27 cu. ft. of iron oxides were removed. Calculations based on the total surface areas indicate a thickness varying from 0.0006 to 0.0031 in. However, in the 13 cases a thickness of 0.001 ±0.0004 in. is true except for treatments 16 and 19.

MR. R. B. DONWORTH.[4]—In giving thought to the mechanism of the evaporation of steam in a boiler tube or more particularly in a furnace wall tube, I think we should go a little further into the physical conditions which may exist where this occurs.

Take a point in a furnace wall tube operating at say 900 psi. where 20 per cent of the water has been evaporated to steam, the steam then occupying more than 80 per cent of the volume. Consider then a pot boiling on the kitchen stove with bubbles of steam coming up through the water. I don't recall ever noticing much difference in the appearance except in the size of the bubbles and the points from which they start. But if the steam occupies 80 per cent or more of the volume, are we sure whether we have bubbles of steam in water or droplets of water in steam? It may make considerable difference which of these conditions prevails. If the fluid is essentially steam containing droplets of water, the inside surface of the tube on the fire side tends to be above the saturated temperature of the water and is peppered continuously by the small droplets of water which bounce around and evaporate to steam, increasing further the volume of steam. Obviously the ratio of steam to water increases greatly as the mixture passes from the bottom to the top of a long vertical furnace tube.

---

[3] The Dow Chemical Co., Midland, Mich.

[3a] The boldface numbers in parentheses refer to the combined bibliography appended to these papers, see p. 940.

[4] Betterment Engineer, Power Stations Dept., Duquesne Light Co., Pittsburgh, Pa.

TABLE V.—BOILER DEPOSITS CONTAINING COPPER.

New York City New York	5% Copper 10% Fe_2O_3 20% Fe_3O_4 10% $Mg_3Si_2O_7 \cdot 2H_2O$ 55% $Ca_{10}(OH)_2(PO_4)_6$	Utility
Atlantic City New Jersey	10% Copper 10% Fe_2O_3 30% $NaAlSi_2O_6 \cdot H_2O$ 20% $(MgFe)_3(SiAl)_4O_{10}(OH)_2 \cdot 4H_2O$ 20% $Ca_{10}(OH)_2(PO_4)_6$	Utility
Perth Amboy New Jersey	20% Copper 10% Fe_2O_3 30% $NaAlSi_2O_6 \cdot H_2O$ 20% $(MgFe)_3(SiAl)_4O_{10}(OH)_2 \cdot 4H_2O$ 20% $Ca_{10}(OH)_2(PO_4)_6$	Chemical
Beaver Falls Pennsylvania	5% Copper 15% Fe_2O_3 5% $Mg_3Si_2O_7 \cdot 2H_2O$ 75% $Ca_{10}(OH)_2(PO_4)_6$	Building
Philadelphia Pennsylvania	10% Copper 45% Fe_3O_4 15% Fe_2O_3 30% $Ca_{10}(OH)_2(PO_4)_6$	Utility
Akron Ohio	5% Copper 10% Fe_2O_3 80% Fe_3O_4 5% SiO_2	Rubber
Toledo Ohio	25% Copper 35% Fe_3O_4 25% $NaAlSi_2O_6 \cdot 2H_2O$ 15% $Ca_{10}(OH)_2(PO_4)_6$	Utility
Comstock Michigan	10% Copper 70% Fe_2O_3 20% $Ca_3Fe_2(SiO_4)_3$	Utility
Flint Michigan	10% Copper 10% Fe_2O_3 10% Fe_3O_4 70% $Ca_{10}(OH)_2(PO_4)_6$	Automotive
Milwaukee Wisconsin	5% Copper 25% Fe_3O_4 75% $4CaO \cdot Na_2O \cdot 6SiO_2 \cdot 2H_2O$	Textile
Chicago Illinois	15% Copper 30% Fe_2O_3 40% Fe_3O_4 15% $4Na_2O \cdot CaO \cdot 4Al_2O_3 2CO_2 \cdot 9SiO_2 \cdot 3H_2O$	Utility
Chicago Illinois	20% Copper 5% Fe_2O_3 50% Fe_3O_4 10% $Mg_3Si_2O_7 \cdot 2H_2O$ 15% $Ca_{10}(OH)_2(PO_4)_6$	Utility
Chicago Illinois	15% Copper 30% Fe_2O_3 40% Fe_3O_4 15% $4Na_2O \cdot CaO \cdot 4Al_2O_3 \cdot 2CO_2 \cdot 9SiO_2 \cdot 3H_2O$	Utility
Freeport Illinois	5% Copper 40% Fe_3O_4 40% $Mg_3Si_2O_7 \cdot 2H_2O$ 15% $Ca_{10}(OH)_2(PO_4)_6$	Manufacturing
Houston Texas	10% Copper 10% Fe_3O_4 20% $Mg_4Si_2O_7 \cdot 2H_2O$ 60% $Ca_{10}(OH)_2(PO_4)_6$	Refinery

If that is the case, the metal is not continuously in contact with the solution as we may have been thinking. Instead, the metal is in an atmosphere of gas with discrete water particles impinging on it. Can galvanic action occur without continuity of the electrolyte? Under this hypothesis, these par-

TABLE VI.—OPERATIONAL CORROSION DATA FROM INHIBITED ACID TREATMENT OF BOILERS FOR EAST COAST UTILITIES.

A. Boiler Treatment	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12	No. 13	No. 14	No. 15	No. 16	No. 17	No. 18	No. 19	No. 20
B. Date boiler was treated	11-6-43	7-28-44	4-14-45	11-13-43	6-27-44	4-20-45	2-26-44	3-10-45	3-2-44	8-2-45	7-25-45	8-15-45	3-23-45	3-30-45
C. Gallonage of acid	16 260	16 750	17 000	13 750	15 800	17 000	33 000	32 650	33 000	9000	9000	9000	17 500	17 000
D. HCl in inhibited acid, per cent	5	4	4.5	5.5	4.5	4.9	3.7	4.0	4.5	6.3	6.3	5.4	4.5	4.35
E. Average temperature, deg. Fahr.	160	170	175	170	170	175	175	165	170	130	135	125	170	170
F. Time in hours (filling, treating, and draining)	7	7	7	7	7	7	10	6	12	12	12	8	7	9
G. Inside surface area exposed, sq. ft.	20 000	20 000	20 000	20 000	20 000	20 000	42 750	42 750	42 750	12 000	12 000	12 000	20 000	20 000
H. Iron in drain acid, per cent	0.49		0.35	0.45	0.31	0.40	0.56	0.30	0.56	0.83	0.61	0.64	1.21	0.33
I. Iron in drain acid 8.3 × line C × line H, lb.	663		497	515	408	567	1540	816	1540	621	458	480	1760	468
J. Iron dissolved due to acid corrosion assumed in absence of ferric iron (assuming corrosion rate of 0.015 lb. per sq. ft. per day), lb.	87.5	87.5	87.5	87.5	87.5	87.5	206	122	248	67	67	45	76	95
K. Iron due to iron oxide solubility and assumed ferric ion corrosion (line I minus line J), lb.	575		409	427	320	479	1336	694	1292	554	391	435	1684	373
L. Iron assumed to be due to ferric ion corrosion (29 per cent of line K, based on 50 per dent Fe_2O_3 and 50 per cent Fe_3O_4), lb.	167		119	124	93	139	388	202	376	161	114	126	488	108
M. Iron due to iron oxide solubility (line K-line L), lb.	408		290	303	227	340	948	492	916	393	277	309	1196	265
N. Iron oxide dissolved (line M × 1.43), lb.	586		415	434	325	486	1360	704	1312	562	396	442	1710	379
O. Iron oxide dissolved (line N ÷ (5.2 × 62.37)), cu. ft.	1.81		1.28	1.34	1.00	1.50	4.20	2.17	4.05	1.73	1.22	1.36	5.27	1.17
P. Thickness of iron oxide on internal boiler surface before treatment (line O ÷ line G × 12), in.	0.0011		0.0008	0.0008	0.0006	0.0009	0.0012	0.0006	0.0011	0.0017	0.0012	0.0014	0.0031	0.0007

ticles of water striking a slightly overheated metal probably cause minute cracking and possibly oxidation or other effect on the slightly cracked tube metal. If this is true, high-pressure furnace wall tubes probably have a limited life under the best of conditions.

MR. R. K. SCOTT[5] (*presented in written form*).—Mr. Corey is certainly to be congratulated on his thoroughness in collecting data and opinions from various sources on the effect of copper and iron oxide deposits in the corrosion of steam generating tubes.

The rôle of copper in corrosion can be studied with better chances of success if a little more is known about the chemistry of the reactions taking place in alkaline solution. Accordingly, we have set out to find first of all what happens to copper compounds when they enter the boiler.

While it is true that copper may be carried along mechanically in a finely divided metallic state, it seems more probable that copper enters the boiler as cuprous (Cu^+) or cupric (Cu^{++}) ion, either dissolved or as finely divided particles of oxide or hydroxide.

We have long considered that metallic copper forms in boilers by reduction of cuprous or cupric salts by hydrogen evolved from the attack on boiler steel. This stand has been completely confirmed in laboratory tests.

In order to investigate what happens to copper compounds in contact with corroding boiler steel, in a reasonable length of time, it was necessary to use fine steel powder instead of a metal surface. This was obtained by cutting boiler steel with a power saw, and using the fraction of cuttings which passed through a No. 100 sieve and was retained on a No. 200 sieve. A weighed portion of the steel powder was then charged into a platinum-lined bomb with a sufficient amount of solution to thoroughly cover the steel and to provide considerable excess. It was found that 20 ml. of solution with 5 g. of steel was a satisfactory working condition. The bomb was closed, connected to a pressure gage and placed in a thermostatically controlled furnace with the gage on the outside. Most of the tests were run at 325 C., corresponding to a pressure of pure water of 1735 psi. Heating was continued for approximately 6 hr. At the end of this time, the bomb was allowed to cool and any residual hydrogen pressure noted.

The products of the corrosion tests were removed from the bomb, washed, and examined by X-ray diffraction.

Most of these tests were run in 5N sodium hydroxide, a concentration which could be reasonably expected when a boiler water concentrates under a layer of deposit. In this solution, if we charge only the steel powder, the reaction goes according to the following equation:

$$3Fe + 4H_2O \xrightarrow{NaOH} Fe_3O_4 + 4H_2$$

Now if we add metallic copper to the bomb charge, the corrosion of steel still goes on according to the above reaction, and the copper is not materially affected. If, however, we add an oxide of copper, that oxide will be reduced by the amount of hydrogen generated. If we start with a small amount of copper oxide, we find in the product only metallic copper, Fe_3O_4, and iron. A residual hydrogen pressure is found only where the corrosion has exceeded the ability of the oxygen of the copper oxide to combine with the hydrogen formed. If more copper oxide is added so that not all of it is reduced to metal, we do not get any residual hydrogen pressure. As a matter of fact, if the amount of copper oxide is slightly in excess of that required to combine with the hydrogen liberated, the magnetic oxide formed during the corrosion is further oxidized to ferric oxide, and we find in the products of the corrosion tests metallic copper, metallic iron, and Fe_2O_3. Further addition of copper oxide can result only in lowering the state of oxidation of the copper. For instance, if excess cupric oxide is used, we find curpous oxide, possibly some unreacted cupric oxide, and the mineral delafossite ($Cu_2O \cdot Fe_2O_3$). This compound is interesting in that it does not particularly resemble magnetic iron oxide structurally, being a hexagonal crystal, yet is somewhat magnetic. It can be readily prepared in a bomb simply by mixing cuprous oxide and ferric oxide in a platinum liner and heating for a 6-hr. period in 5N sodium hydroxide. To my knowledge, this compound has not been previously reported in boiler deposits. It has been identified in our laboratory as a minor constituent in one or two instances in operating boilers, usually associated with hard, adherent deposits of hematite (Fe_2O_3) and cuprite (Cu_2O).

---

[5] Research Engineer, Hall Laboratories, Inc., Pittsburgh, Pa.

In general, it may be stated that it makes no difference what copper compound enters the boiler. If hydrogen is being generated in any point in the system, reduction of the copper compound takes place in that area, and copper or a lower oxide will probably be found as a deposit in that vicinity. Copper oxide will be found only where insufficient hydrogen is generated to complete the reduction to metallic copper.

We cannot, at this time, make a general statement as to whether or not copper increases the rate of corrosion of boiler steel. We have, however, found in a number of tests that an increased amount of hydrogen was generated in the presence of metallic copper over that which formed without the copper.

I doubt whether dynamic studies of these reactions will tell us much, since the conditions cannot be readily duplicated where the picture is complicated by the flow of liquids and gases around the solid particles. In order to be able to reproduce these tests with any reasonable precision, all the conditions other than those which are deliberately varied must be held within very narrow limits. I believe that any data involving dynamics would best be obtained from an operating boiler, which is a much better integrator of variables in flow than any piece of laboratory equipment.

MR. A. H. REYNOLDS.[6]—Most of the presentation and the discussion has centered around the effects of copper or copper compounds which are unintentionally carried into the boiler by the feedwater and are deposited as scale or sludge. The general assumption prevails that copper so introduced is cathodic with respect to the steel boiler or the tubes upon which it might be deposited.

The paper contains at least one reference in the form of a report from an operating company to the Edison Electric Institute which pointed out that "there is evidence that metallic copper is anodic to steel, as attested by occasional reports that copper plugs, gaskets, and ferrules in contact with steel in economizers and drums corrode rapidly, particularly where the ratio of the exposed area of the copper to that of the steel with which it is in contact is relatively small."

We have had considerable experience with corrosion problems resulting from the use of copper ferrules in boilers in contact with a steel header and steel tubes. It is frequently found that the copper ferrule is attacked and gradually destroyed, while little or no effect is observed on the steel header and tubes. This action on the copper ferrule might lead to the assumption that the ferrule is anodic to the steel, but on the other hand, our inability to find attack on the steel surfaces could hardly in itself justify the conclusion that the steel was protected, because of the much greater surface over which corrosive action would take place if the steel were anodic to the copper.

---

[6] Directing Chemist, Dearborn Chemical Co., Chicago Ill.

Inspection of copper ferrules removed from boilers after being subjected to corrosive attack as indicated above show a surprising condition. Instead of a more or less uniform corrosive action on the portion of the ferrule in contact with the boiler water, the action is limited to the circular end of the ferrule which projects from the header into the water. Two possible explanations of this localized attack are presented. The first explanation is that a capillary space exists between that portion of the copper ferrule extending into the boiler water, and the boiler tube which it surrounds (assuming a fire tube type boiler). Boiler water can concentrate in this capillary space until it reaches an alkalinity high enough to have direct chemical solvent action on the copper ferrule. If such is the case, the attack would be on the inner surface of the ferrule or at the end of it, where it would be in contact with the concentrated solution.

The second explanation runs parallel to the first one in that it assumes the capillary space and the concentration of boiler water in this space. Having a common metal, copper, in contact with solutions of decidedly different concentration would establish a concentration corrosion cell. The corrosive action would be most severe in the area where the solutions of different concentration meet, or in other words, at the circular end of the ferrule.

Simple galvanic action with the copper ferrule anodic to the steel header and tube would not seem to explain the localized action on the end of the ferrule and the absence of such action where the ferrule is tightly squeezed between the header and the tube. Neither would it explain the lack of corrosive action on the cylindrical surfaces of the ferrule exposed to the boiler water, particularly in view of the low ratio of exposed copper to exposed steel.

Mr. Donworth in his discussion suggested that in a boiling solution, steam may be the continuous phase adjacent to the heating surface. We have taken a series of high-speed moving pictures of boiling solutions and the heating surfaces which produce them, at approximately five thousand frames per second. Running these films at normal speeds has permitted us to slow down the action and observe the manner in which steam bubbles are formed. A study of these films makes it clear that in almost all cases of steam generation, which we observed, water is the continuous phase adjacent to the heating surface.

It is only with very pure waters or specially treated waters and with very high heat input that film boiling is approached.

MR. B. J. CROSS.[7]—One characteristic of the type of corrosion discussed by Mr. Corey in vertical furnace wall tubes is the very restricted local nature of the attack. Quite frequently only a 6 to 12-in. length of tube is affected, the areas above and below the point of attack being in almost perfect condition.

There is no consistency in the location within the furnace. The corroded area may be at a point where high rates of heat transfer may be expected and they also have been found where the heat input is known to be low. This would appear to eliminate as primary causes both excessive heat input and inadequate circulation. Metallurgical examination of the tube metal at the points of corrosion does not indicate that the metal has been overheated except in instances where the accumulation of products of corrosion has been great enough to interfere with the transfer of heat.

---

[7] Manager, Research Department, Combustion Engineering Co., Inc., New York, N. Y.

Corrosion has occurred where local metal stresses are suspected, such as at welds and bends and, in the same boiler, at sites remote from such locations.

It may be pointed out that this type of corrosion is peculiar to furnace wall tubes where one side is exposed and the other side protected from the heat. This must, of course, set up thermal stresses in the tube wall. However, this is a general condition throughout the furnace and in itself would not account for local attack.

This inconsistency in the location of points of attack would seem to indicate some more general cause than any of those considered in the foregoing comment.

It is suggested that some type of more-or-less porous deposit is necessary to provide a focus of attack. Such local deposits have been found at welds and tube bends. They also may be provided by periodic oxygen attack forming even an incipient tubercle, under which corrosion may continue in absence of oxygen.

Corrosion has been found under weld spatter in resistance welded tubes and also under metal lamina where the weld has been poorly broached.

If such deposits can be postulated it would not be difficult to account for the type of corrosion noted, and the problem would then be resolved to the investigation of the many ways by which deposits may form and means of their prevention.

MR. M. D. BAKER.[8]—Mr. Corey has prepared an excellent summary of the papers that have described the problem of corrosion in high pressure steam generators. The contradictory opinions expressed by fifteen investigators regarding the effect of copper, copper oxides, and iron oxides on the rate of corrosion shows the need for basic data that will determine the cause and mechanism of this type of corrosion. After these have been determined, then prevention can be obtained. All information relative to this type test of corrosion should be collected and studied, after which an intelligent program can be instituted.

An enlargement of some of the statements which were made in our paper (**14**) which Mr. Corey mentioned may clarify our position and explain why we think the presence of copper has no connection with the cause or rate of corrosion.

Copper and copper oxides have always been present in the high-pressure boiler deposits. The varying percentage of copper in the deposits has had no relation to the amount of corrosion that was found. Copper gaskets are used to seal the hand caps. These gaskets have proven satisfactory except for one case when they failed after the gaskets had given satisfactory service for a period of 18 months. After acid cleaning, new gaskets were placed throughout the entire boiler. In some headers, in less than 30 days, the attack on the gaskets had been so severe, that replacement was necessary. In all cases of failure the copper disappeared and no evidence was found that the boiler metal was being attacked.

The explanation for the copper gaskets failure is that a cell had been established between the copper and the iron with the copper being anodic. Prior to acid cleaning, chemical analyses indicated that the boiler metal was coated with a film of sulfite-phosphate compound. This film prevented any electrolytic action between the copper and the iron. Acid cleaning removed this film. After acid cleaning, about one year elapsed before the presence of this film could again be detected. After the film had again been formed by the sulfite and phosphate used in boiler water treatment, copper gaskets were satisfactory for use in the boilers. Those gaskets have now given over two years of service with no indication of failure.

---

[8] Chief Chemist, West Penn Power Co., Springdale, Pa.

The presence of metallic copper between the laminations of the corrosion barnacles is explained by the fact that copper oxide is always present in the boiler deposits. Boiler water that seeps in carries copper oxide into the space between the laminations. Here the higher temperatures and electrolysis causes the water to break down into hydrogen and oxygen. The oxygen accelerates corrosion, the hydrogen reduces the copper oxide to copper also penetrates the tube metal causing the embrittlement of the metal. Metal attack and barnacle formation have been completely arrested by reducing the amount of dissolved oxygen in the boiler feedwater and by carrying in the boiler water excess sulfite of 2 to 10 ppm.

The need for research to determine how to prevent corrosion in high-pressure steam generators is very great. Mr. Corey has started the study by his paper. From this point some group, possibly the Joint Research Committee on Boiler Feedwater Studies should continue the research program.

MR. J. B. ROMER.[9]—As Mr. Corey points out, the question of whether deposits of copper and iron oxide have a specific effect in causing certain types of failure in high-pressure steam generators is one of vital importance to boiler operators, manufacturers and water treatment specialists.

Mr. Corey is quite right in stating that the literature does not contain any data based on experiments under controlled conditions of heat transfer, fluid temperature, velocity, as well as the composition and physical condition of the metal surface.

To furnish such data adequately, one must conduct the experiments under conditions that closely approach those existing in high pressure boilers. Data from experiments run at and below 212 F. cannot be extrapolated to 600 F.

About 25 years ago I was called in to investigate a serious case of corrosion occurring in a boiler operating at 200 psi. Briefly, the history was several years of corrosion-free operation and then suddenly a bad case of corrosion had developed. Investigation showed large quantities of copper, and the only remedy applied was that of eliminating the source of the copper, and as far as I know further trouble has not occurred on this particular job. Between then and the present time I have encountered a number of cases where copper has suddenly appeared; corrosion has followed and the basic remedy was to get rid of the copper. In those days residual oxygen was frequently present and presumably at higher levels than is true today.

Those interested in this subject want more than circumstantial evidence in order to settle this question and this is as it should be.

We do not know what the pH of the boiler water is at operating temperatures. We consider that if the pH at room temperature is at certain limits we will have a safe condition at operating temperatures. Likewise we do not know what the polarity is between the boiler metal and the deposits at operating temperatures, and we may well have a reversal in polarity at operating temperature compared with room temperature. A local acid condition may exist even though the general conditions are alkaline.

---

[9] Research and Development Dept., The Babcock & Wilcox Co., Alliance, Ohio.

I have seen a great deal of copper in certain boilers and no visible evidence of any harmful effects; however, the form in which the copper has been present in these boilers is different than in those cases where corrosion or intergranular disintegration have occurred. In the latter case the copper, either as the metal or a mixture of metal and oxide, has been well mixed with iron oxides, either as hard barnacles or masses of sludge.

In the article by Brister and Romer (3), which was referred to by Mr. Corey, we set forth a number of conditions which we felt might be involved. Apparently Mr. Corey is referring to the fourth condition wherein we referred to a galvanic couple which creates anodic conditions beneath the barnacles, etc. It would appear from reading Mr. Corey's paper that the term "barnacles" was unfortunate as he apparently considered that a barnacle must be the result of an action forming a pit whereas we have seen the same condition underneath loose masses that were quite wet and full of copper and iron oxide. In those cases where a lot of copper has been present and no corrosion evident, the copper has generally been quite hard and dense, frequently plated onto the tube or in the form of "ropes" at given locations.

From the fact that elimination of copper in many jobs has stopped the trouble, I definitely believe the copper is in some way involved, but it is not necessarily the only factor. I quite agree that sound experimental evidence based on research is needed to clarify this situation.

MR. J. H. FOOTE.[10]—Mr. Corey's excellent paper serves to bring to our attention an aspect of the boiler corrosion problem which sometimes escapes us: namely, that the concurrence of a multiplicity of conditions and factors contributing to corrosion are generally present, and therefore no single solution can be ascertained, much as one might desire it.

Not only may numerous factors apply to the situation existing in the boiler as a whole or in any part of it, but intense localization of conditions may produce such puzzling situations as exist in a boiler tube cited to have experienced severe Fe_3O_4 scaling within inches of clean surface. Such situations puzzle us because we tend to visualize a uniformity or simplicity of conditions which evidently do not exist, even along a single tube of a steam generator.

It has been our practice to recognize this multiplicity of factors contributing to corrosion and to attempt solution of the problem by systematically attacking each factor in turn. Especially where high-pressure condensing turbine-generator units are involved, we study the elimination of elements and factors one by one.

For instance, if free oxygen could be eliminated from the boiler water, corrosion may be less likely to occur. Therefore oxygen should be removed from the water in the boiler and then prevented from entering the system at any point once it has been eliminated. So instead of causing the free oxygen to combine with the chemicals introduced into the boiler, which may conceivably add new factors, we take the oxygen out in the condenser, paying especial attention to condenser and hot well design so that the oxygen is encouraged to leave the water and can be swept away. Then through careful design of valves and pumps together with alert operation and maintenance of joints, glands, and packing, every care is taken to keep oxygen from entering the system. Even should this not constitute a complete solution to the oxygen problem, it certainly is in the right direction.

---

[10] Su[illegible]ing Engineer, The Commonwealth & Southern Corp[illegible]kson, Mich.

Then we may find ammonia to be present and consider the best place to remove it from the system. Ammonia dissolves readily in cool water. Why not take it out of the system at a cool point? So by wasting only a few BTUs through the wasting of the condensate from the inter-cooler and after-condenser just beyond the vacuum jet we get rid of the ammonia as well as the air.

Of course, tubes must be clean, and we have found experimentally that Dowell cleaning is effective—in addition to oxygen and ammonia removal, etc.

Design and operation of the boiler and burners may contribute mightily to the factors producing localized corrosion. We discover that these localized spots of corrosion are more often than not associated with intense release of heat in localized areas. The solution may involve amelioration of several contributing factors such as: (*a*) increase the capacity of down-comers suplying these tubes with water, (*b*) provide segregation or other facilities to guard against pirating by adjacent tubes, (*c*) examine the possibilities of flow reversal at varying loads, (*d*) examine flame pattern to guard against impingement, (*e*) examine operating practice with respect to wall cleaning and deslagging through the use of wall blowers; etc., etc.

Thus, we see that a great variety of contributing factors participated in by the chemicals in the water, by the design of the equipment, and by its operation all collaborate in producing troublesome as well as desirable results.

It is evident that Mr. Corey has performed a significant service in presenting this list of eight difficulties and seven possible solutions. Let us all study the boiler corrosion problem as a concurrence of many factors operating some independently and some sequentially. Perhaps we may add to Mr. Corey's list as our understanding of these problems and their solutions develops.

MR. F. R. OWENS[11] (*presented in written form*).—Since our discussion of deposits of copper and copper oxide in boilers (**22**), we have encountered hundreds of similar deposits, with tube failures in only very few cases. However, we continue to lack factual data in spite of numerous plant studies; hence we must continue to express a speculative opinion.

Our studies during the past eight years have not changed our conclusions (**22**) of 1940. We do not believe that copper or its oxides is a fundamental cause of corrosion in high-pressure steam generators. However, it must be recognized that the equilibria involved in high-pressure steam generators is quite complex and may be easily disturbed through some slight change in either physical or chemical environment. Hence, it is entirely possible in such instances that copper or copper oxides may become a secondary factor in the corrosion cycle.

We are entirely in accord with Mr. Corey in that the subject requires an intensive research effort—in addition to the scope suggested by the author it is our opinion that the liberation of hydrogen as first reported by Hecht (**16**) merits early attention. Also, we are reminded of our very limited factual data on equilibria and suggest that McKinney's (**21**) effort might well be extended.

MR. J. M. DECKER.[12]—Deposits in the boilers of the Detroit Edison Co. contain from 3 to 35 per cent copper, some of which exists as the oxide, and from 42 to 78 per cent iron oxide. Available analytical data seem to indicate that the ratio of copper to iron oxide is higher in boilers operating at pressures of 600 psi.

---

[11] Secretary, Cyrus William Rice and Co., Inc., Pittsburgh, Pa.

[12] Research Dept., The Detroit Edison Co., Detroit, Mich.

and higher than in boilers operating at lower pressures.

It has been our experience that iron oxide and copper deposits do not cause damage in boilers unless they form in locations where high rates of heat transfer occur. These deposits usually do not cause the bagging-tube type of failure such as that caused by water-scale deposits in tubes. Usually failure is caused by corrosion beneath the deposit which progresses until the metal is perforated or until the thinned metal in the corroded area cracks open.

Designing boilers so that water circulation is rapid enough to prevent the deposition of sludge from the boiler water in areas where heat-transfer rates are high aids in preventing failures caused by sludge deposits. In cases where trouble has been encountered, a design change to increase the rate of circulation is often very helpful.

At Delray Power House, two, long, crooked tubes forming part of the side wall of a flue gas by-pass zone corroded badly under a deposit of a sludge in a section near the mud drum that was exposed to the radiant heat from the furnace. This condition was found in three boilers of identical design. To correct the condition, the two corroding tubes, which were located between the mud drum and the steam drum, were connected within the mud drum with a steel box arrangement to two nearby generating tubes that discharged into the mud drum from the back water wall. The outlet ends of the corroding tubes were extended through the baffle plates in the steam drum beneath which they normally discharged. This was done to reduce resistance to circulation in the tubes. As a result of the two changes, the rate of circulation in the tubes was increased. Sludge no longer accumulates in the area where corrosion formerly occurred.

Keeping the amounts of sludge in boilers at a minimum also helps to prevent damage. Much can be done to accomplish this by corrosion-preventive water treatment and by shielding, with erosion-resistant alloys, tubes in feed-water heaters and condensers that are exposed to erosive action of steam and water.

MR. A. A. BERK.[13]—Whirl has already mentioned the occurrence of zinc in the deposits. We rarely find deposits of carbuncles, or sludge for that matter, that do not contain some zinc. It may be small but there always is some. We already have speculated on a local reversal of pH with regard to corrosion in low-pressure boilers. Such reversals from alkaline to acid have been reported in pits. Soluble zinc compounds could form under such conditions and cause rapid pitting, as magnesium chloride was known to do in some of the old boilers operated with untreated waters.

My second thought is that to get rapid pitting there usually must be hydrogen depolarization? Scott pointed out that copper salts can react with hydrogen very rapidly; could not copper, therefore, be an excellent depolarizer, and would not that account for the copper spotted around carbuncle areas? It has been pointed out by Reynolds, and also by Baker, that copper may go into solution in boiler water under certain circumstances. In other words, solid metallic copper can be dissolved by boiler water. When dissolved copper reaches the points at which carbuncles are growing, it could depolarize hydrogen at the surface and pitting could be accelerated. Boiler-water concentrations affect the ionic state of copper in the boiler; we should investigate to understand the chemistry involved and to create trouble-free conditions of operation.

MR. R. C. COREY (*author's closure*).—

---

[13] Supervising Chemist, Boiler Water Research Section, U. S. Dept. of the Interior, Bureau of Mines, College Park, Md.

One of the main purposes of this paper, to stimulate discussion from those who may have information that would help to clarify the questions regarding copper and iron oxides in boilers, has been amply fulfilled, and the author is deeply grateful to the discussors for their contributions.

Mr. Whirl raises two interesting points regarding copper: the possible effects of small amounts of zinc and nickel in the copper matrix, and of the state of subdivision of the copper. Since other nonferrous metals are known to occur in copper deposits, which Mr. Clark's discussion illustrates so well with mention of finding up to 30 per cent of zinc in Naval boiler deposits, this factor cannot be ignored. Nor can the state of subdivision of the copper, which ranges from extremely finely divided particles embedded in magnetite to a continuous film or sheet, be eliminated from consideration.

Mr. Clark believes the metallic copper frequently found adjacent to damaged areas is the result and not the cause of corrosion—an opinion that is shared by many others.

Mr. Alquist's analyses, particularly those of deposits from boilers on various services, which are given in Table V, afford a valuable background for research, especially so when complete information on the sources of the samples are made available. It would be desirable to know the boiler type, pressure and rating; the type of feedwater and makeup; the nominal boiler water analyses; the location in the boiler where the samples were taken; and many other factors that might possibly be correlated with the analyses.

Mr. Donworth considers the possibility that the steam-water ratio in a steaming tube may be so high that the mixture is essentially a single phase, and therefore insufficient water is available for galvanic action. Since the quality of *over all* mixture in a tube, in zones where the kind of attack under consideration takes place, generally is relatively low, there is probably enough liquid phase for electrochemical action to occur if other conditions are favorable. But experiments (5a) on heat transfer under forced circulation, at 500 to 3300 psi., have demonstrated that under certain conditions of heat input to a steaming tube and the mass velocity of fluid in the tube, a *localized* single phase zone, referred to as a steam blanket, may occur. The entire matter, however, of the dynamics of a steaming tube under operating conditions still is one of inference, since no reliable direct observations of the water-side of such a tube have been made. This points to another factor that must be considered in connection with studies of the type under discussion.

Mr. Scott's interesting comments, based upon preliminary experimental work, demonstrates that under conditions believed to simulate closely those of a steaming tube, metallic copper can be produced from one of its oxides. His remark that more hydrogen was generated in the bombs when copper was present implies intensification of attack by metallic copper. The extreme significance of this observation prompts the author to suggest that Mr. Scott extend his experiments over a wider range of conditions, and determine if the platinum liner plays some part in the corrosion mechanism. Mr. Scott is to be commended for his reserve in drawing conclusions from the data he has thus far accumulated.

Either explanation for the attack of copper ferrules in boilers, offered by Mr. Reynolds, is valid, depending upon conditions; direct caustic attack where concentrations may become very high, or crevice type of corrosion in capillary spaces. The author has seen the exposed portions of copper ferrules in steel-tubed economizers corrode at a rapid rate under conditions where neither steaming nor concentration effects could occur. Thus, it would appear that under some conditions of boiler operation the possibility that copper may be anodic to steel cannot be dismissed.

Mr. Cross has put his finger on one of the most common, yet most baffling, aspects of furnace wall-tube corrosion—the randomness of the attack. In a wall with fifty or sixty tubes, severe attack may take place in only a few, scattered tubes; adjacent ones being unaffected. There is no reason to believe that the fluid velocity and the heat input are appreciably different at a given elevation in adjacent tubes, yet one tube may fail due to deep, oxide-filled pits and those on either side be in good condition. The author subscribes wholly to Mr. Cross' suggestion that local deposits and pits formed by dissolved oxygen during outages, may, during normal operation, become points where boiler salts can concentrate and result in rapid attack.

Mr. Baker's comments, based on personal experiences with tube damage in high-pressure steam generators, are valuable for their objectivity. He, also, believes the metallic copper to be the result rather than the cause of corrosion.

Mr. Romer corroborates a factor mentioned in the paper: cases where units have operated for a number of years with none of the type of corrosion damage under discussion, and then severe attack occurred. A complete history of operating conditions prior to the failures is absolutely essential in cases of this kind; unfortunately, however, plant records of water conditions frequently are sketchy or non-existent. If Mr. Romer is correct in attributing the cause of corrosion to copper, a question naturally arises: Was the copper present in the boiler for a long time prior to the tube failures, with slow but progressive attack, or did the copper deposit suddenly as the result of a change of operating conditions, and cause corrosion in a relatively short period?

Mr. Foote has outlined several factors that must be considered in the over-all problem of internal corrosion: oxygen, ammonia, internal cleanliness of tubes, flame impingement, and fluid circulation defects. Again, it must be emphasized that case histories are essential if tube damage is to be correlated with any of these factors.

Mr. Decker's experience indicates that corrosion occurs beneath sludge deposited on the tubes, and suggests that circulation should be rapid enough in areas of high heat transfer to keep solids in suspension. There have been many efforts in this direction, but generally it is considered that high fluid velocities are not the final answer to the problem of preventing sludge deposits on tubes, particularly in vertical runs. Reducing the sludge entering the boiler to a minimum, as Mr. Decker suggests, would appear to be the most logical step.

COMBINED BIBLIOGRAPHY

(1) F. N. Alquist, "A Geographic Study of Deposits and Corrosion," Paper presented before National Association of Corrosion Engineers, St. Louis, Mo., April, 1948. (To be published in N.A.C.E. *Corrosion.*)

(2) W. D. Bissell, B. J. Cross, and H. E. White, "Special Studies of the Feedwater-Steam System of the 2000 psi. Boiler at Sommerset Station of Montaup Electric Company," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, No. 4, pp. 429–442 (1946).

(3) P. M. Brister and J. B. Romer, "Corrosion and Oxidation Experiences in High Pressure and High Temperature Steam Service," *Transactions*, Electrochemical Soc., Vol. 91, pp. 655–680 (1947).

(4) W. W. Cerna and R. K. Scott, "Water Conditioning for the 2000 psi. Boiler at the Sommerset Station of Montaup Electric Company," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, p. 429 (1946).

(5) R. C. Corey, "Internal Corrosion of Furnace Tubes of High Pressure Boilers," *Combustion*, March, 1947, p. 39.

(5a) W. F. Davidson, P. H. Hardie, C. G. R. Humphreys, A. A. Markson, A. R. Mumford, and T. Revese, "Studies of Heat Transmission Through Boiler Tubing at Pressures from 500 to 3300 Pounds," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, pp. 553–591 (1943).

(6) Edison Electric Institute Publication P-10, "Boiler Auxiliaries 1946–1947," p. 10

(7) Edison Electric Institute Publication M-6, "Boiler Auxiliaries 1944–1945," p. 1.

(8) Edison Electric Institute Publication P-8, "Turbines, Condensers and their Auxiliaries (1945–1946)," pp. 31–34.

(9) Edison Electric Institute Publication M-4, "Boilers and Combustion 1944."

(10) H. Gaunt, "Copper Deposits in Boilers," *Electrical Times*, Vol. III, pp. 523–524 (1947).

(11) E. W. Gillham and A. E. Howes, Discussion of paper by D. Parkhouse, "Copper Deposits in Boilers," *Electrical Times*, Vol. III, p. 339 (1947).

(12) A. J. Gould and U. R. Evans, "Some Experiments on Corrosion of Steel in Boiler Water," *Journal*, Iron and Steel Inst., Vol. 155, pp. 195–200 (1947).

(13) R. Habib, "An Unusual Case of Corrosion in High Pressure Boilers," *Chaleur et Industrie*, Vol. 19, pp. 512–514 (1938).

(14) L. E. Hankison and M. D. Baker, "Corrosion and Embrittlement of Boiler Metal at 1350 Pounds Operating Pressure," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 69, pp. 479–486 (1947).

(15) C. A. Hawkins and H. L. Solberg "Corrosion by High Temperature Steam," *Corrosion Handbook*, Electrochemical Soc., John Wiley and Sons, New York, p. 511 (1948).

(16) Max Hecht, Private Communication published, F. M. Speller "Corrosion Causes and Prevention," Table LXV, p. 424, Second Edition, McGraw-Hill Book Co., New York, N. Y.

(17) Max Hecht, E. P. Partridge, W. C. Schroeder and S. F. Whirl, "Boiler Corrosion," *Corrosion Handbook*, Electrochemical Soc., John Wiley and Sons, New York, N. Y., p. 523 (1948).

(18) Ibid, p. 520.

(19) J. A. Keeth, "Some Corrosion Problems Encountered in Steam Plant Operations," *Corrosion*, Vol. 2, No. 2, June, 1946, pp. 101–123.

(20) E. H. Mitsch and B. J. Yeager, "Experiences with Internal-Boiler Surface Corrosion in 1450 lb. Open-Pass Boilers at West End Station of the Cincinnati Gas and Electric Company," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 69, pp. 487–491 (1947).

(21) D. S. McKinney, "The Calculation of Equilibria in Dilute Water Solutions," *Proceedings*, Am. Soc. Testing Materials, Vol. 39, p. 1191 (1939).

(22) F. R. Owens, "Influence of Copper with Respect to Corrosion in Boilers," *Combustion*, May, 1940, pp. 41–44.

(23) D. Parkhouse, "Copper Deposits in Boilers," *Electrical Times*, Vol. III, p. 196 (1947).

(24) G. U. Parks, W. S. Patterson, and W. F. Ryan, "Operating History and Performance of a 2000 psi. Forced-Circulation Boiler at Sommerset Station of Montaup Electric Company," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, p. 411 (1946).

(25) F. G. Straub, "Wall-Tube Corrosion in Steam-Generating Equipment Operating Around 1300 psi.," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 69, pp. 493–499 (1947).

(26) L. Tronstad and R. Veimo, "Preliminary Researches on the Action of Water in Copper Pipes," *Journal*, Inst. Metals, Vol. 66, pp. 17–32 (1940).

(27) S. F. Whirl, *Public Service* (Employee publication of the Philadelphia Co.), August, 1938.

(28) H. A. Wagner, J. M. Decker, and J. C. Marsh, "Corrosion-Erosion of Boiler Feed Pumps and Regulating Valves," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 69, No. 4, pp. 389–403 (1947).

(29) E. H. Wyche, L. R. Voight, and F. L. LaQue, "Corrosion in Crevices," *Transactions*, Electrochemical Soc., Vol. 89, p. 149 (1946).

(30) Uhlig, "Corrosion Handbook," p. 520 (1948).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON METALLOGRAPHY IN COLOR

Numerous requests have been received by Committee E-4 on Metallography from those working in this field for the proper techniques to use to record photographically the characteristic colors that are seen visually. The committee, therefore, decided to sponsor a symposium on Metallography in Color through its Subcommittee IV on Photography. The symposium, consisting of the five papers listed below, was presented at the First Session of the Fifty-first Annual Meeting of the American Society for Testing Materials held in Detroit, Mich., on June 21, 1948.

Metallography in Color—R. P. Loveland (reprinted from ASTM BULLETIN, No. 128' May 1944. Not presented at the Annual Meeting session)
Microscope Optics for Color Metallography—J. R. Benford
Quality and Quantity of Illumination in Color Metallography—R. P. Loveland
Color Metallography Simplified—Mason Clogg, Jr.
Some Applications of Color Metallography—W. D. Forgeng
The Application of Color Photography to the Study of Nonmetallic Inclusions—A. M. Hall and E. E. Fletcher

These papers together with discussion will be issued as A.S.T.M. Special Technical Publication No. 86, entitled, "Symposium on Metallography in Color."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MAGNETIC TESTING

The Symposium on Magnetic Testing was sponsored by Committee A-6 on Magnetic Properties and presented at the Third and Fourth Sessions of the Fifty-first Annual Meeting of the American Society for Testing Materials held in Detroit, Mich. on June 22, 1948.

The rapid development of new and superior magnetic materials in recent years has given rise to new and often difficult problems in connection with magnetic testing. Furthermore, there has been continued activity and development in the field of magnetic analysis by which the condition and quality of materials are determined from the results of non-destructive magnetic tests. It is the purpose of this symposium to indicate certain of the problems encountered in magnetic testing and to outline some of the methods that have been devised to overcome them. The following ten papers were presented:

Testing Magnetic Materials—B. M. Smith
Permanent Magnet Testing Methods and Their Validity in Determining Product Performance—C. A. Maynard and J. E. Mitch
Core Loss Test for Narrow Silicon Steel Strip—J. A. Ashworth
D-C. Permeability Testing of Epstein Samples with Double-Lap Joints—D. C. Dieterly
A New D-C. Permeameter—W. J. Carr, Jr.
Variation of Core Loss and Permeability of Electrical Grade Silicon Sheet Steel—A. C. Beiler and P. L. Schmidt
The Evaluation of Hysteresis Core Loss by Power Equations—Horatio W. Lamson
Magnetic Analysis Inspection in the Steel Industry—Theodore Zuschlag
Magnetic Stress Analysis—P. E. Cavanagh and T. Wlodek
The Testing of Magnetic Recording Media—D. E. Wiegand

The papers together with discussion are issued as A.S.T.M. Special Technical Publication No. 85, entitled, "Symposium on Magnetic Testing."

THE SPECTROCHEMICAL ANALYSIS OF CEMENTS AND OTHER MINERAL PRODUCTS*

By M. F. Hasler,[1] C. E. Harvey,[1] and F. W. Barley, Jr.[1]

Synopsis

The chief objects of the investigation were to study and extend the principal methods of spectrochemical analysis which have been proposed for the analysis of non metallic samples. Of special interest was the development of simpler and improved methods of quantitative analysis for portland cement.

A semi-quantitative method has been extended to cover materials of general interest to the cement industry such as cement, limestone, clay, iron ore, and sand. A simple, reasonably accurate, D-C. arc method, employing specially shaped electrodes, has been devised for the analysis of a group of elements in portland cement: aluminum, magnesium, iron, manganese, titanium, sodium, and potassium. A more elaborate method employing briquets of cement mixed with graphite has been modified to minimize the effects of a variation of physical and chemical form in samples, and to increase the sensitivity of the potassium determination. The main problems of spectrochemical analysis of nonmetallic samples are discussed against the background of a study of the principal methods of analysis proposed to date for solid samples.

Spectrochemical methods of semi-quantitative and quantitative analysis are now available for use in the cement industry. These methods allow a six- to eight-fold increase in speed of analysis and in the case of aluminum, sodium, and potassium, an appreciable increase in accuracy over wet chemical methods.

In the manufacture and use of cement, there are many instances when simple, rapid methods for obtaining the equivalent of accurate chemical analyses can be used to great advantage. In addition to the problems related to raw materials, including the original prospecting for these materials and the routine testing of them for production control, there is the routine testing of the final products of manufacture, clinker and cement, to insure quality control. In the use of cement in the making of concrete, analysis of the mineral aggregates, and the water employed, can often be of value in assuring the production of an adequate final product.

Samples usually consist of indeterminate mixtures of complex compounds containing many elements. Largely because of this indeterminateness, no simple methods of chemical analysis are available which insure accurate, detailed results for materials of this type. This in turn has greatly slowed down the development of other methods of analysis

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Director of Research, Chief Spectrographer, and Spectrographer, respectively, Applied Research Laboratories, Glendale, Calif.

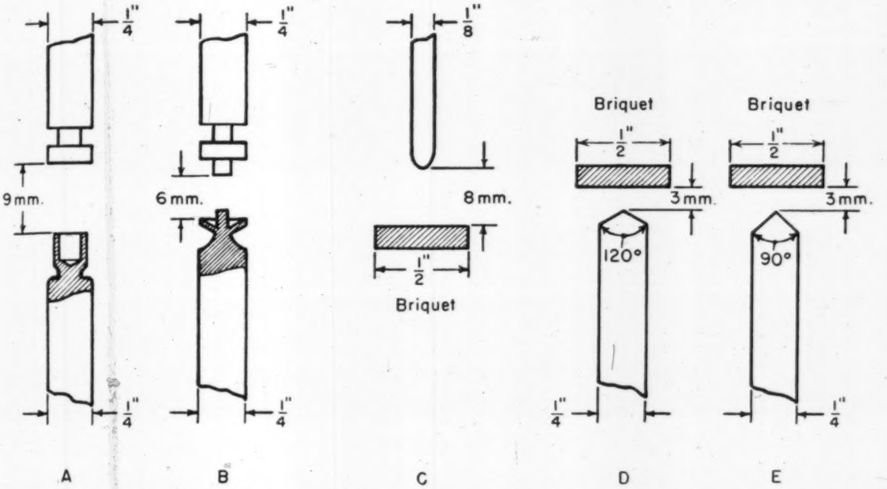


FIG. 1.—Types of Electrode Arrangements Employed for the Spectrochemical Analysis of Non-metallic Samples.

which are based on reference to standards of accurately known composition. Among such methods, the spectrochemical one is by far the most powerful, providing as it may, qualitative and quantitative analyses of some seventy elements by employment of a few direct procedures.

It is the purpose of this paper to describe a few of these procedures which have been found by our methods-research division to be most generally applicable in the field of nonmetallic sample analysis. The analysis of portland cement is utilized as an example to illustrate many of the points involved in connection with the accurate quantitative analysis of nonmetallic samples. Basic data relating to clays, limestones, sands, etc., are also provided.

The simplest, most universal procedure is first considered, followed by more complex ones of greater accuracy but of more limited scope. The descriptions of the various procedures are followed by a brief general discussion of the instrumentation required.

SEMI-QUANTITATIVE SAMPLE ANALYSIS

Method:

The method developed by C. E. Harvey (**1**)[2] for semi-quantitative spectrographic analysis is a very useful procedure for survey analysis work. It consists of the vaporization in a d-c. arc, of 10 mg. of powdered sample mixed with 10 mg. of S.P. (National Carbon Co. Special Purity) No. 2 graphite placed in a graphite cup electrode of the type shown as (*A*) in Fig. 1. The intensities of certain lines of each of the elements detectable on the spectrogram are compared to the intensities of the backgrounds adjacent to these lines. These intensity values are converted to percentage composition by the formula:

$$C = 2k\left[\frac{I\ (\text{line plus background})}{I\ (\text{background})} - 1\right]$$

where C is the concentrarion, k is a factor

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 958.

TABLE I.—OPERATING CONDITIONS FOR SEMI-QUANTITATIVE SAMPLE ANALYSIS.

1—Upper electrode	¼-in. high purity graphite rod as shown in Fig. 1*a*, negative polarity
2—Lower electrode	¼-in. high purity graphite rod as shown in Fig. 1*a*, positive polarity
3—Electrode spacing	9 mm. maintained during arcing by adjustment
4—Alignment	Electrodes spaced symmetrically above and below optical axis
5—Optics	5-in. focal length cylindrical lens placed 10-in. away from primary slit of spectrograph, focuses arc image on grating
6—Source	D.c. Arc adjusted to 13 amp. on short circuit giving approximately 10 amp. while arcing
7—Exposure time	Arc to complete vaporization of sample and electrode cup – about 90 sec.
8—Spectrograph	Original grating, 6.9A/mm. dispersion
(*a*) Slit width	60μ
(*b*) Spectrum regions utilized	2380A–4600A; 4580A–5900A. Used simultaneously with dual region attachment
9—Photographic Emulsion	Eastman Spectrum Analysis No. 2
10—Development	2 min., 65 F., Eastman formula D-8 2 parts stock solution to 1 part water

for a particular spectrum line, and I is the intensity of the line or background as indicated. Table I lists the conditions of analysis. Table II lists the spectrum lines and the constants, *k*, for elements of likely interest in a variety of materials encountered in the manufacture and use of cement.

Standards:

The published semi-quantitative tables are directly applicable to laboratories having instruments whose dispersion and freedom from scattered light are comparable to the Applied Research Laboratories' 1.5-m. Spectrographs used for establishing the factors. Standards are not essential in such cases, although they are useful in checking instruments and extending the method. Five standard powders are available covering various groups of elements. These may be added to any pure matrix material or to a sample of known composition such as a Bureau of Standards' sample, to provide standard samples. Analysis before as well as after addition is recommended to check the purity of the matrix material. Since in these cases the concentrations of the various elements are known, the above mentioned formula can be used to calculate the constant, *k*.

Results:

By the use of this method and with the data of Table II, a spectrographic laboratory can be in a position to give semi-quantitative analyses of any samples of this type, in about 30 min. per sample. These analyses should not differ by more than 30 to 50 per cent from the true values. Obviously with this limited accuracy the analyses can only be used for exploratory or survey work, where it is important to define the problem rather than to obtain the exact analysis of a given sample. However there are many such problems in connection with the raw materials used in the production of both cement and concrete, which can be rapidly solved by the method.

In this connection the determination of alkalies, particularly sodium and potassium, is of great importance. Though these elements are included in the semi-quantitative method, their sensitivity, particularly in the case of potassium, is not all that could be desired. Thus where these elements are of particular concern, the method described in the next section is recommended because of both increased sensitivity and accuracy.

QUANTITATIVE ANALYSIS WITH THE D-C. ARC

The direct-current arc has certain features as a spectrochemical source, not shared by any other convenient thermal or electrical source. The most important one in this respect is the ability to convert a large portion of the energy utilized in the arc, into thermal energy at the electrodes. This energy is of such a magnitude that it is a relatively easy matter to volatilize completely any normal sample of mineral origin, placed in the anode. This is the basis of excitation used in the semi-quantitative method described in the previous section. However, it can be refined to produce more accurate quantitative results without undue complication. This improvement results from two features, use of a center-post platform electrode and employment of an internal standard element. Often the internal standard can be the major constituent, when it remains fairly constant in percentage from sample to sample.

Method for Refractory Elements Without Fluxing:

The analysis of cement has been worked out as a typical example of a use of this method. Samples of cement are prepared rather carefully, as only a 4-mg. portion is used to make an analysis. A 1-g. portion of commercially ground cement is ground further in an agate mortar until it will all pass through a No. 325 screen. A 20-mg. portion of this is mixed with an equal portion by weight of

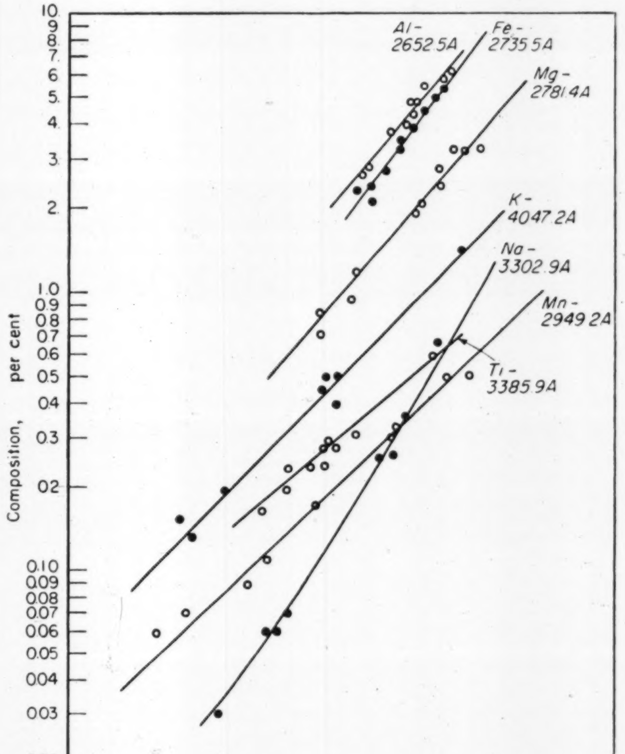


Fig. 2.—Working curves for D-C. arc, Platform-Electrode Analysis, Showing Intensity Ratio of Spectrum Lines Indicated to Internal Standard Lines, as a Function of Percentage Composition.

TABLE II.—FACTORS FOR SEMI-QUANTITATIVE ANALYSIS.
(X indicates interference)

Element	Spectrum Line, Å	Cement	Limestone	Clay	Iron Ore	Sand
Aluminum	2575	0.10	0.012	PC	0.074	0.03
	2568	0.16	0.021	PC	X	0.04
Antimony	2598	X	0.014	X	X	0.07
	2878	0.092	0.053	0.048	0.051	0.12
Arsenic	2780	X	0.29	X	0.21	0.12
	2860		0.34	0.21	0.23	0.18
Barium	3072	0.18	0.12	0.20	0.14	0.34
Beryllium	3130	0.0011	0.0005	0.0005	0.0005	0.00027
Bismuth	3068	0.0022	0.0011	0.0013	0.0009	0.0013
Boron	2498	0.0079	0.005	0.006	X	0.018
	2497	X	0.009	0.009	X	0.02
Cadmium	3261	0.031	0.022	0.008	X	0.0075
Calcium	4227	PC	PC	0.0002	X	0.00023
	3179	PC	PC	0.07		
	3007	PC	PC	0.12		
Chromium	4254	0.00084	0.0005	0.0007	0.00045	0.00043
	4340		0.011	0.022	0.016	
Cobalt	3454	0.00095	0.0016	0.001	0.0009	0.00058
	3044	0.014	X	0.011	0.015	0.01
Columbium	3163	0.033	X	0.022	0.017	0.01
	3094	0.035	0.015	X	X	0.014
Copper	3248	0.00042	0.00005	0.00007	X	0.00004
	3274	0.00091	0.00008	0.0002	0.00009	0.00005
Iron	3021		0.0023	0.0015	PC	0.0018
	2838	0.88	0.40	0.42	PC	0.50
Lead	2833	0.018	0.013	0.017	0.017	0.042
	2577		0.10	0.20	0.11	X
Lithium	3233	0.040	0.016	X	0.016	0.0073
	2741	0.15	0.054	0.09	X	0.079
Magnesium	2852		0.00009	0.00015	X	0.0003
	2781	0.52	0.20	0.05		0.13
Manganese	2798	X	0.001	0.002	0.0062	0.003
	2933	0.062	0.026	0.02	0.025	0.03
Mercury	2537	0.21	0.063	0.085	X	0.30
Molybdenum	3133	0.0020	0.0013	0.0033	X	0.0028
Nickel	3415	0.0023	0.0015	0.001	0.0007	0.00048
	3316		0.033	0.023	0.011	0.027
Phosphorus	2536	X	0.14	X	X	X
	2553	0.77	0.34	0.45	X	X
Potassium	4047	X	0.48	0.37	0.80	0.17
	3446	1.5	1.4	1.5	X	1.0
Silicon	2881	PC	0.009	PC	0.0025	PC
	2435	PC		PC	0.19	PC
Silver	3281	0.00024	0.00014	0.00013	0.0002	0.0001
Sodium	3302	0.049	0.045	0.04	0.06	0.033
	2853	X	0.28	X	X	0.80
Strontium	3464	0.08	0.07	0.025	0.07	0.03
Tantalum	3013	0.18	0.13	0.08	X	0.11
Tin	3175	0.0043	0.0025	0.0025	0.0024	0.0028
	2422		0.045	0.054	0.062	X

TABLE II.—*Continued.*

Element	Spectrum Line, Å	Cement	Limestone	Clay	Iron Ore	Sand
Titanium	3373		0.0033	0.001	0.0009	0.00061
	2942	X	0.008	0.005	X	0.045
Tungsten	2944	0.11	0.10	0.07	X	0.083
Vanadium	3184	X	X	0.0016	0.0013	0.0022
	2952	0.057	0.045	0.037	0.048	0.04
Zinc	3345	0.069	X	0.037	0.025	0.015
	3076	X	0.18	X	X	0.10
Zirconium	3392	0.0072	0.0077	0.0035	X	0.0005
	2571		0.014	0.017	0.014	X

S.P. No. 2 graphite. An 8-mg. portion of this mixture is placed on the platform electrode. The conditions of analysis are identical with those indicated in Table I except that the electrodes and gap have the form and dimension shown in Fig. 1(*B*) and a current of 10 amp., measured by short-circuiting the electrodes, is employed. Also in this work the visible range of the spectrum is not utilized and the electrodes are not adjusted during exposure. The spectrum lines used, and the range of concentrations for each element as well as the precision and the accuracy obtained, are shown in the upper portion of Table III. Precision is based on the reproducibility obtained on eight repeat analyses using a single arcing for each; accuracy is based on the difference between chemical and spectrographic results where the latter is based on the average obtained from duplicate arcings on a group of 21 standard cements. In making calculations, the working curves shown in Fig. 2 were utilized. For the most precise results, a correction for variations in the calcium content is made. This correction is based on the fact that the intensity of the calcium lines used as internal standards vary linearly with concentration. Therefore intensity ratios must be multiplied by a factor which is the calcium content of the sample divided by 64 per cent. These corrected intensity ratios are applied directly to the working curves of Fig. 2 to obtain the various percentage compositions.

TABLE III.—RESULTS FOR DC. ARC, PLATFORM-ELECTRODE ANALYSIS OF CEMENTS.

Elements Analyzed	Spectrum Lines, Å		Range of Analysis (as oxides) in per cent	Precision Standard Deviation, per cent	Accuracy, Average Concentration Difference (per cent of Sample)
	Elements Analyzed	Internal Standard			
		REFRACTORY OXIDES			
		(Calcium)			
Aluminum	2652.5	3150.7	2.8 to 6.1	2	0.24
Magnesium	2781.4	3150.7	0.7 to 3.6	3	0.14
Iron	2735.5	3150.7	2.1 to 5.4	5	0.20
Manganese	2949.2	3150.7	0.06 to 0.50	4	0.014
Titanium	3385.9	3286.1	0.16 to 0.30	7[a]	0.021
		ALKALI OXIDES			
		(Lithium)			
Sodium	3303.0	3985.8	0.03 to 1.2	5	0.02
Potassium	4047.2	3985.8	0.04 to 1.4	7	0.04

[a] Probably high due to laboratory being contaminated with TiO$_2$.

Standardization:

As indicated in the introduction, adequate standards are one of the principal stumbling blocks in the development of comparison methods of nonmetallic sample analysis. Our laboratories were fortunate in obtaining, from the Portland Cement Association Fellowship at the National Bureau of Standards, small quantities of the 21 standards referred to in the work of Helz and Scribner (**2**). This work provided both spectrographic and chemical values so that these standards are of an unusually high order of excellence. Realizing that until such standards are generally available, other means of standardization should be provided, the following was developed: synthetic samples were prepared from the oxides of silicon, aluminum, iron, magnesium, manganese, and titanium, and the carbonates of calcium, sodium, and potassium. Heating of all materials to 600 C. before weighing is recommended. Portions of these standards were briquetted and heated to 1200 C. for 30 min. in holes of graphite blocks which were covered with graphite plugs. Weighing before and after indicated that all the carbonates were decomposed and that fairly stable samples had been formed. These were carefully ground to pass a No. 325 sieve and then analyzed in the regular manner. In all cases, after correction was made for quantities in the blank, the synthetic standards gave values which fell on the curves within the accuracy shown in Table III. This is very remarkable in view of the fact that the highly refractory oxides of aluminum and magnesium were used in the preparation of the standards rather than a standard clay, for instance. This attests to the remarkable properties of the d-c. arc in minimizing the physical and chemical differences which undoubtedly exist between the cement samples and the synthetic samples. These differences will be apparent in subsequent sections. Before closing this discussion of standards it should be emphasized, however, that the standard mixtures cannot be used successfully in the d-c. arc with this method without pretreatment by firing as indicated.

Method for Alkalies:

As may be seen from the above, the complete vaporization of the sample and the use of calcium as an internal standard provide acceptable analyses for the more refractory oxides. However, arcing until complete vaporization of the sample occurs furnishes considerable background, particularly in the region of the potassium lines, while the vapor pressures of calcium compounds are so different from those of sodium or potassium that calcium would be a very poor internal standard.

These facts led to the development of a carrier-distillation method based on the use of lithium carbonate. A 90-mg. sample of cement without graphite is mixed with 10 mg. of lithium carbonate. A 10-mg. portion of this mixture is placed on a platform electrode. The sample is vaporized with the d-c. arc under the same conditions as specified previously in this section, except for 10 sec. only. The lines recommended and the results obtained are shown in the lower portion of Table III.

The lower limit of measurement is particularly low for alkali determinations using these ultraviolet and violet lines. This procedure is recommended for the detection and measurement of low alkali contents—with the exception of lithium of course—in any type of nonmetallic material. Depending, as it does, upon volatilizing the alkalies before any appreciable quantity of the base material is volatilized, results should be reasonably independent of the type of matrix provided it has a sufficiently high boiling point and does not react too rapidly with lithium carbonate. However, standardization with synthetic samples employing the proper matrix is recommended. Such samples should, in general, not be fired as there is the possibility of losing the alkalies by this treatment. Unfired portions of the synthetic cement standards mentioned above, when corrected for carbonate content present, gave values which coincide very well with the working curves established from the analyzed samples.

Method Employing Fluxing While Arcing:

Though the analysis of cement with the d-c. arc using synthetically prepared standards was successful without employing fluxing, there are many cases in the analysis of nonmetallic samples in which that would not be the case. One example is the analysis of clay which is high in aluminum as well as silicon. An analysis of a mixture of oxides which simulates the composition of clay in every detail will not provide the same intensity ratios between the lines of the various elements as is obtained from the clay, when analyzed with the d-c. arc. Aluminum in particular will behave in a different manner in the two samples. This is undoubtedly related to the large difference in volatility between alumina and various aluminum silicates. A heat treatment of the synthetic undoubtedly would improve this situation. However, there is another approach which is quite effective, that is dilution of the sample with lithium carbonate. By mixing nine parts of lithium carbonate with one of sample, and using a 10-mg. sample of the mixture on a platform electrode of the type shown in Fig. 1(*B*), the differences between synthetic and natural samples vanish upon arcing them to completion. Also the dilution of the sample allows the major constituents, silicon and aluminum, to be determined by a comparison of the intensities of their lines to the intensities of appropriate lithium lines. Thus the method is very versatile.

The principal drawbacks of this system are: the small amount of sample utilized, 1 mg.; the impurities added by the lithium carbonate; and the reduction in the limit of detection of elements in the sample due to the large dilution needed. In connection with the second, a simple method of purifying lithium salts is available in the resin cation exchanger. By passing a solution of c.p. lithium chloride through such an exchanger, the impurity content can be lowered to a very small value. The chloride is then converted to the carbonate by treatment with ammonium carbonate (7.—Seventh Quarterly Report).

Method Employing Fluxing Prior to Arcing:

In order to insure the reduction of samples and standards to a common standard form, to increase the amount of sample, and to reduce the amount of lithium carbonate used, a fluxing operation prior to arcing can be utilized. Combining such an operation with d-c. arcing has provided accurate analysis of some of the most refractory types of nonmetallic samples. It is a method to be recommended in the more difficult cases of analysis.

The usual method of fluxing, as employed by the chemist, is designed primarily to put samples in such a form that they will be readily soluble in acid. It is not designed to provide a solid sample uniform in composition throughout, and which can be handled readily. A method for doing this, which has been devised for this work, depends upon dispersing a mixture of sample and flux through a graphite structure composed of flakes of graphite. This is done in practice by mixing one part of sample, one part of flux, and two parts of National Carbon Co. S.P. No. 1 flake graphite together and then briquetting at high pressure. This briquet is then placed in a hole in a graphite graphite block and covered with a piece of graphite rod which fits the hole loosely. It is heated to a high temperature for a definite time. Temperature and time are chosen such that reaction between the sample and flux will occur but excess fluidity will not result. Inspection of the briquet will indicate whether any beads have formed on the surface. Temperature and time should be adjusted to be just less than that required to form beads. Lack of beads will insure uniformity of material within the briquet, as it indicates that no excessive migration of material has occurred. Such briquets can be readily crushed and a 10-mg. portion used for arc analysis. Typical temperatures and times are 1200 C. for 30 min. for highly refractory materials such as some titanates and zirconates, and 900 C. for 1 hr. for less refractory materials such as portland cement, etc.

Conclusions Based on Experience with the d-c. Arc:

The d-c. arc has been used for many years for the qualitative anzlysis of a wide variety of materials. Though several investigators (**3, 4, 5**) have indicated means for its use quantitatively, it has not enjoyed the wide acceptance that the spark method has had, for instance, in the analysis of metals. Actually there are many applications, particularly in the field of nonmetallic analysis where the d-c. arc allows a simple straightforward method to be developed which is almost as effective as much more elaborate methods. This should be kept in mind in evaluating the methods to be discussed in the next sections.

QUANTITATIVE ANALYSIS WITH THE INTERRUPTED D-C. ARC

The great success obtained by the use of interrupted discharges, such as sparks and a-c. arcs, and solid electrodes in the field of metal analysis, has led several investigators to attempt to use interrupted discharges and briquetted specimens for nonmetallic sample analysis. Three of the most important contributions in this field (**2, 6, 7**) make use of S.P. No. 1 flake graphite as the binding material to produce, upon mixing with a powdered sample, flat briquets, $\frac{1}{2}$ in. in diameter. A discharge is passed to these briquets by a counter electrode of high purity graphite. Three arrangements of these electrodes are shown in Figs. 1(*C*), (*D*), and (*E*).

Extensive studies using this "point to plane" technique have indicated that the major problem is not in obtaining adequate precision on repeat analysis of the same sample but rather in obtaining adequate accuracy upon intercomparing samples of known composition. This indicates that the differences in physical and chemical form, from one sample to another, are the chief sources of error. The effects, however, are of a different order of magnitude than those mentioned in connection with the preparation of standards for the d-c. arc method. In that case a mixture of oxides and carbonates sintered at 1200 C. served to provide adequate standards without fluxing for the analysis of cement. Such standards used without fluxing are entirely inadequate for the type of analysis indicated above. With these limitations clearly in mind a discussion of the methods can be undertaken.

Method of Analysis with Briquets but without Fluxing:

The work of Helz and Scribner (2) referred to previously is an outstanding example of this method. It employs an overdamped 60-cycle d-c. discharge with arc-like characteristics to analyze briquets produced from a mixture of cement sample, cobalt oxide, potassium nitrate, and S.P. No. 1 graphite. It represents an improvement over the d-c. arc method, described above, in two important features. All of the determinations can be made at one time with a single procedure, and a relatively large sample of cement is employed—0.5 g.—which makes for smaller relative errors from contamination. However, the actual amount of material being vaporized into the discharge is probably not greatly in excess of that vaporized in the d-c. arc method, hence sampling errors are not greatly reduced. The precisions for iron, manganese, and titanium are somewhat greater than with the d-c. arc method. This is probably due, in part, to the use of cobalt oxide as an internal standard, in part to the better control of the discharge. In the case of the determination of aluminum and magnesium, or of sodium and potassium, however, there is little difference between the two methods in precision or accuracy.

Method of Analysis with Briquets and with Fluxing Prior to Discharge:

The chief criticisms of the above method are its sensitivity to the exact physical and chemical form of the sample, which is not serious as long as well processed cement is being analyzed, and the lack of sensitivity for potassium, which makes for large errors in low percentage determinations. It was chiefly to improve these conditions that a modification of this method was developed. It utilizes a one-to-one dilution with lithium carbonate, a fusion as indicated in the section entitled "Method employing fluxing prior to arcing" under the discussion of "Quantitative Analysis with the d-c. Arc," then regrinding and rebriquetting after fusion. Also the polarity of the discharge has been reversed making the sample positive so that more local melting results at the point of discharge.

These things have proved to be effective in reducing the differences resulting from variation in crystal form from sample to sample. This fact is clearly demonstrated in the work reported by the authors to the Signal Corps (7) in which the analysis of various alkaline earth titanates has been discussed. The more obvious benefits of this addition of lithium carbonate were an increase in the ratio of potassium line intensity to background intensity by a factor of two and the elimination of the effect of potassium concentration on the sodium determination as reported by Helz and Scribner (2). The latter resulted from the stabilizing effect of a high lithium content on the temperature of the discharge. The increased sensitivity to potassium allowed determinations down to 0.2 per cent potassium oxide to be made without the addition of potassium nitrate and below this with an addition of this amount.

Operating conditions are shown in detail in Table IV while Table V indicates the spectrum lines employed, the range of analysis, and the precision, and accuracy achieved. Precision is based on eight repeat analyses of single briquets; accuracy is based on the comparison between spectrographic and chemical values for twenty-one standard samples, averaging the results obtained from the analysis of two briquets. In general the precision results are comparable to those reported in (2) though

TABLE IV.—OPERATING CONDITIONS FOR INTERRUPTED D-C. ARC CEMENT ANALYSIS

1—Sample preparation	0.250 g. of sample, 0.250 g. of lithium carbonate, 0.025 g. of cobalt oxide, 0.50 g. of S.P. No. 2 graphite mixed (for low potassium determinations add 1.07 mg. of potassium nitrate). Briquet at 1600 lb. total pressure on ½-in. diameter plunger. Fuse at 900 C. for 1 hr. Regrind and rebriquet at 8500 lb. total pressure.
2—Lower electrode	Cylindrical briquet ½-in. in diameter; positive polarity.
3—Upper electrode	Graphite rod ⅛ in. in diameter rounded at one end.
4—Gap	8 mm. initial setting, unaltered during exposure.
5—Alignment	Top of pellet 3 mm. below the optical axis.
6—Optics	Same as Table I except quartz diffusion screen replaces arc in position and arc is moved back 2.5 in. Thirty per cent filter is used in front of camera to intercept the 3082A aluminum line.
7—Source	Multisource unit supplying over-damped discharges.
(a) Capacitance	45μf.
(b) Inductance	400μh.
(c) Resistance	40 ohms
(d) Phase angle	30 deg.
(e) Output potential	940 v.
8—Exposure	30 sec. on each side of briquet successively to produce single spectrogram.
9—Intensity measurements	Correct for background where appreciable on spectrogram.
10—Other features	As shown in Table I.

aluminum, sodium, and potassium seem somewhat improved. Improvement in the aluminum determination is probably largely due to comparing its line intensity to that of calcium rather than to cobalt. Just as was done in the arc work, intensity ratios to calcium are corrected proportionately for calcium content. The working curves for the various elements are shown in Fig. 3.

Standards:

The synthetic standards, sintered at 1200 C. without flux and used successfully in the d-c. arc work, were analyzed by the last method described. Systematic deviations between these standards and the analyzed samples indicated the lack of true equivalence of the two types of material. It also indicated that the fluxing action in the discharge was not sufficient to eliminate the differences involved. Since it is necessary to standardize spectrographic methods daily if the highest degree of accuracy is to be maintained, it is necessary either to obtain standard cements for this purpose or to standardize a set of typical cement samples. The best way to do the latter, on the basis of the work reported here, would be by the d-c. arc method, comparing samples which are being standardized with synthetic standards. Averages of analysis in quadruplicate should provide very acceptable standardization.

Doubtless, by starting with standard clays, limestones, iron ore, etc., and heating sufficiently to sinter before fluxing, something approaching a standard cement could be produced. However on such a basis the compounding

TABLE V.—RESULTS FOR INTERRUPTED DC. ARC, BRIQUET ANALYSIS OF CEMENTS.

Element Analyzed	Spectrum Lines, Å		Range of Analysis (as oxides) in per cent	Precision Standard Deviation, per cent	Accuracy Average Conc. difference (per cent of Sample)
	Elements Analyzed	Internal Standard			
		(Calcium)			
Aluminum	3082.2	3006.9	2.8 to 6.1	2	0.2
		(Cobalt)			
Magnesium	2779.8	3072.3	0.7 to 3.6	2	0.1
Iron	2973.3	3072.3	2.1 to 5.4	2	0.2
Manganese	4034.5	3072.3	0.06 to 0.50	3	0.01
Titanium	3998.6	3072.3	0.16 to 0.30	4[a]	0.02
Sodium	3302.3	3072.3	0.03 to 1.2	3	0.02
Potassium	4044.1	3072.3	0.04 to 1.4	6	0.03

[a] Probably high due to laboratory being contaminated with TiO_2.

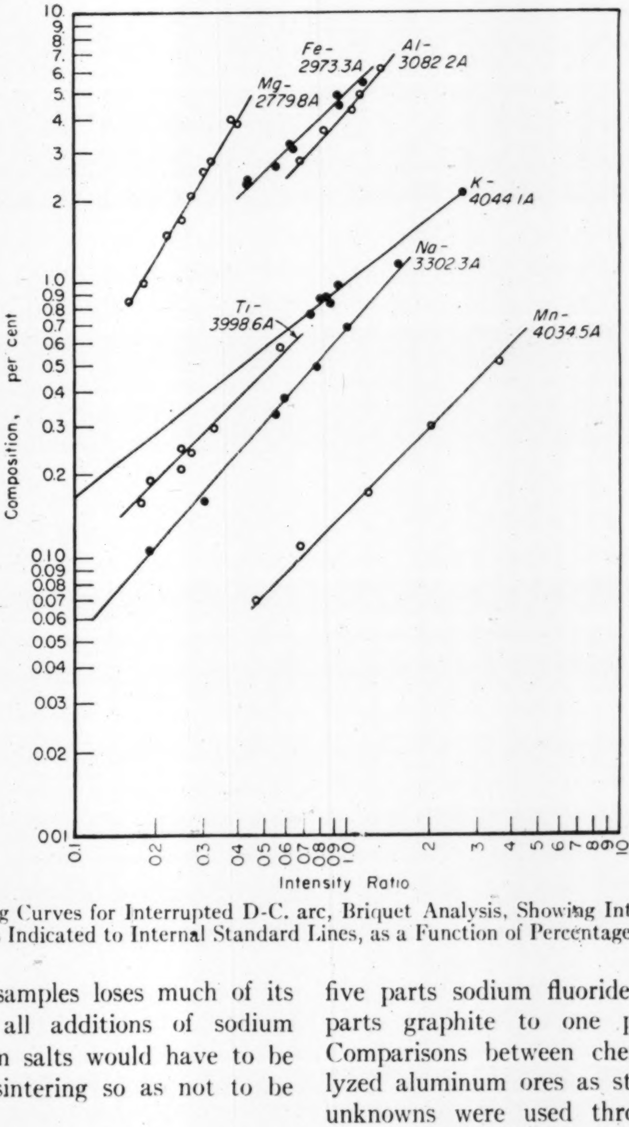


Fig. 3.—Working Curves for Interrupted D-C. arc, Briquet Analysis, Showing Intensity Ratio of Spectrum Lines Indicated to Internal Standard Lines, as a Function of Percentage Composition.

of synthetic samples loses much of its value. Also all additions of sodium and potassium salts would have to be added after sintering so as not to be volatilized.

Quantitative Analysis with the High-Voltage a-c. Spark

The work of Churchill and Russell (**6**) demonstrated the possibilities of using a high-voltage spark for the analysis of briquets of nonmetallic samples. In their work the sample was diluted eighteen-fold by adding a mixture of five parts sodium fluoride and twelve parts graphite to one part sample. Comparisons between chemically analyzed aluminum ores as standards and unknowns were used throughout this work. Hence the approximate equivalence of standards and samples was assured.

A similar method of analysis was tried on cement samples using the fourfold dilution used in the previous section with one part lithium carbonate and two parts graphite for each part sample. A standard a-c. spark unit furnishing 120 strokes per sec. was employed.

A 0.007 μf. capacitance charged to 16,000 v. is discharged through an oscillating circuit of 360 μh. inductance and residual resistance to form the spark discharge. The electrode system shown in Fig. 1(*d*) was utilized. Using these conditions of analysis, all of the refractory oxides in cement were determined with a precision which averaged 5 per cent. Since these results are somewhat poorer than those obtained by the previous method and since the accuracy appeared considerably less, the exact lines used and the working curves will not be given at this time.

This bears out the findings discussed in the earlier report (**7**) that the more arc-like discharges tend to minimize the differences between samples due to differences in crystal form and the more spark-like discharges tend to enhance these differences. Use of more inductance and capacitance such as referred to in the paper by Churchill and Russell (**6**) would tend to make the discharge more arc-like as would the use of more alkali flux. Successful analysis of cement and other mineral products could probably be achieved by changes of the above procedure along these lines. The problem becomes essentially one of determining how much dilution of the sample can be utilized without losing all of the lines of a given element at a given concentration in the background spectrum. The one-to-one dilution of sample with lithium carbonate mentioned above is already too great for the lower sodium and potassium determinations desired in cement. This presents a dilemma which can only be resolved by analyzing those elements, the lines of which are lost by dilution, by another method which is more sensitive. Use of the a-c. arc in combination with the a-c. spark offers this possibility.

Before closing this section on use of spark-like discharges for nonmetallic sample analysis, it might be well to point out that similar results have been obtained with an oscillating discharge obtained with the multisource using 2 μf., 50 μh. and 0.4 ohms.

QUANTITATIVE ANALYSIS WITH THE A-C. ARC

As indicated above, the a-c. arc offers the possibility of detecting and measuring low percentages in briquetted samples of nonmetallic material. It offers some special problems when alkali fluxes are utilized, as the arc does not then extinguish itself properly between half cycles of the applied voltage. An electrode system such as indicated in Fig. 1(*c*) becomes useless, as the arc readily climbs up the small rod-like upper electrode. The most satisfactory results have been obtained with the electrode system shown in Fig. 1(*e*). The flat briquet on top and the more sharply pointed electrode below keeps the discharge centered fairly well though even then occasionally it may run over to the edge of the briquet.

The a-c. arc employed was a new type of spark-ignited unit with a 600-v. secondary, operated at 3 amp. in the arc circuit. It provides results equivalent to a standard high-voltage a-c. arc operated at the same current, but allows the use of larger gap spacings.

Using this system of analysis and the briquets composed of one part of cement sample, one part of lithium carbonate, and two parts of S.P. No. 1 graphite, precision values of 6 per cent were obtained for sodium and potassium using eight single repeat analyses. Excellent sensitivity resulted with this arrangement, and the accuracy appeared quite acceptable for regular cement samples. However, the results with synthetic samples were quite at variance with those from natural samples. This was quite different from the experience with both the ordinary d-c. arc and the interrupted arc as in both these cases synthetic and standards behaved similarly for the measurement of sodium and potassium. The exact reason for this is not too clear except that this type of discharge does not appear to localize itself very much even microscopically, but appears to travel continually over the sample. This may limit local fusion which appears to be a necessary prerequisite for successful comparison of synthetic standard and sample.

INSTRUMENTATION

To assist the reader who is unfamiliar with spectrographic methods, the six steps involved in an analysis are described, together with the necessary apparatus. Step one has to do with sample preparation. In the case of the d-c. arc methods, this step would involve only simple equipment, such as special electrode cutters, besides the usual grinding and weighing apparatus found in most laboratories. For the more elaborate methods, a furnace allowing temperatures up to 1200 C. to be utilized continuously is required as well as a briquetting press providing pressures of at least 8500 lb. total pressure to be applied on a $\frac{1}{2}$ in. diameter mold. Step two is the excitation of the sample. This requires either a simple d-c. arc unit or a Multisource unit,[3] the latter providing either the interrupted d-c. arc or the continuous arc as required. Step three is the photography of the spectrum. This requires a spectrograph providing adequate dispersion at the potassium line 4044.1 so as to give a good ratio of line to background. A 1.5-m. grating spectrograph providing a dispersion of 7A per mm. throughout the spectrum was utilized in the work described. Step four is the development of the spectrogram under controlled conditions by means of a thermostated developing machine. Step five is the identification of the various lines of interest in the spectrogram and the measurement of the transmission value of each line. A comparator-densitometer allowing these operations to be performed simultaneously is utilized for this step. Step six is the final calculation of the percentage composition of the sample. This is done by means of a calculating board which converts the transmission readings as obtained on the densitometer to percentage composition. All of this equipment, with the exception of the furnace, is standard spectrographic equipment available on the commercial market. A suitable furnace may be obtained from most laboratory supply houses.

CONCLUSIONS

This work represents a study of the application of accepted spectrochemical methods to the analysis of portland cements and other mineral products. A semiquantitative method is adapted for these types of materials. Two methods of quantitative analysis are presented in detail, one employing a platform electrode and a d-c. arc, the other, sample briquets and an interrupted d-c. arc. In both cases accurate analyses for the oxides of aluminum, magnesium, iron, manganese, titanium, sodium, and potassium have been developed. These quantitative methods provide results which are comparable in accuracy to wet chemical methods for the higher ranges, and more accurate for the lower ranges of concentrations. However the number of man-minutes per analysis is reduced by a considerable factor for the spectrochemical methods as compared to the wet chemical.

---

[3] Applied Research Laboratories' instruments were used for all phases of this work.

In the light of this study and the work of Helz and Scribner (2), it can be stated with assurance that spectrographic procedures will allow the laboratories of cement plants and other interested parties to use well developed techniques which will give consistent results of high accuracy. Methods are not complicated and can be performed by laboratory personnel with the aid of adequate spectrographic equipment.

In conclusion, recognition should be given to the Signal Corps Engineering Labs., for fostering the development of spectrochemical methods for the analysis of nonmetallic samples through a research contract with our laboratories. Though none of this cement work was done under this contract, some of the methods utilized were developed under it (7).

REFERENCES

(1) C. E. Harvey, "Semi-Quantitative Spectrographic Analysis," (285 pgs.)—Published by Applied Research Laboratories, Glendale, Calif. (1947).

(2) A. W. Helz and B. F. Scribner, "Spectrographic Determination of Minor Elements in Portland Cement," *Journal of Research*, Nat. Bureau Standards, Vol. 38, RP 1786, p. 439 (1947).

(3) E. K. Jaycox, "Spectrochemical Analysis of Ceramic and Other Nonmetallic Materials," *Journal*, Optical Soc. Am., Vol. 37, p. 162 (1947).

(4) R. W. Smith and J. E. Hoagbin, "Quantitative Spectrographic Analysis of Ceramic Materials," *Journal*, Am. Ceramic Soc., Vol. 29, p. 222 (1946).

(5) M. F. Hasler, C. E. Harvey, and H. W. Dietert, "Quantitative Spectrographic Analysis of Stainless Steels," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 102 (1943).

(6) J. R. Churchill and R. G. Russell, "Analysis of Aluminous Ore by means of Spark Spectra," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 17, p. 24 (1945).

(7) M. F. Hasler and C. E. Harvey, "Eight Quarterly Reports—Squier Signal Laboratory, Signal Corps Contract No. 36-039-sc32254," (Sept. 1946–June 1948 incl.). Distributed by Department of Commerce, Washington, D. C.

DISCUSSION

Mr. W. J. McCoy.[1]—There is no doubt considerable interest in this method as applied to the determination of alkalies because of the problem of alkali-aggregate reaction in concrete. Most of the data presented in the paper was in regard to the determination of alkali in portland cement. Portland cement is an acid-soluble material and there are two other rapid methods—the flame photometer and the A.S.T.M. alternative method—which are applicable only to acid-soluble materials. However, if this spectrographic method could be applied to the rapid analysis of clays and shales and other argillaceous materials not normally acid-soluble, it would be especially valuable. My question is: Can the alkali content of materials not acid-soluble be determined by the same rapid spectrographic procedure as described by the author for portland cement?

Mr. W. C. Hanna[2] (*by letter*).—This report seems to represent some important additions to the pioneering work of Helz and Helz and Scribner on the spectrographic analysis of cement, and extension of the same methods to cement raw materials.

Helz' original method, reported in 1945, was limited to the determination of alkalies in cement by direct current arc. The d-c. arc has the advantages of (1) relative cheapness, (2) simplicity, (3) sensitivity to low concentrations, and (4) high energy, permitting ready volatilization.

Because this method had the disadvantages of "(1) requiring red-sensitive plates which tended to be not uniform, (2) the difficulty of handling small samples in powdered form, and (3) the requirement of additional procedures for lithium and high-potassium concentrations," Helz and Scribner used an interrupted d-c. arc from a multi-source unit in their investigation reported in 1947. They developed a procedure which permitted the determination of aluminum, iron, magnesium, potassium, sodium, manganese and titanium at once by the use of a cobalt internal standard in a sample briquetted in 9½-in. diameter mold at about 8500 lb. total load. They obtained precisions (probable error of a single determination) within 3 per cent of the oxide concentration for aluminum, iron, magnesium, manganees and titanium, 5 per cent of the Na_2O concentration, and 8 per cent of the K_2O concentration. The accuracy (average concentration difference between spectrographic and chemical determinations in per cent of sample) ranged from 0.25 per cent on Al_2O_3 down through 0.14 for MgO, 0.12 for Fe_2O_3, 0.06 for K_2O, 0.02 for Na_2O, and 0.01 per cent for Mn_2O_3 and TiO_2.

By the use of a special "platform" electrode and the use of calcium and lithium as internal standards, Hasler and his associates have been able to extend the use of the d-c. arc to the determination of aluminum, magnesium, iron, manganese, and titanium and improve the determination of the alkalies. The alkalies must still be run separately, however, since arcing to completion gives too much spectral background. A more complicated sample preparation (furnace treatment) may be required for some raw materials. Results are comparable to those of Helz and Scribner.

---

[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

[2] Chief Chemist, California Portland Cement Co., Colton, Calif.

Hasler and his associates have also modified Helz and Scribner's method, using briquets (or pellets) and the interrupted d-c. arc, in an effort to reduce variations due to its extreme sensitivity to the exact physical and chemical form of the sample and to increase the sensitivity for potassium. This involves diluting the sample with an equal amount of lithium carbonate flux, briquetting, fusing at 900 C. for 1 hr. (cement), regrinding and rebriquetting, as well as reversing the polarity to make the sample positive. Precision obtained was within 2 per cent of the oxide concentration for aluminum, magnesium and iron, within 3 per cent for manganese and sodium, and 6 per cent for potassium. Accuracy ranged from 0.2 per cent for Al_2O_3 and Fe_2O_3 through 0.1 per cent for MgO, 0.03 per cent for K_2O, 0.02 per cent for TiO_2 and Na_2O, to 0.01 per cent for Mn_2O_3.

These are the best results to date, but greater precision and accuracy would be desirable for potassium and greater accuracy for aluminum and iron.

We are sorry that the authors have given no more specific data on the time required for an analysis or the total number of determinations which can be made in a day by the various methods. Obviously the purchase of such expensive equipment by an average cement plant would be nearly impossible to justify except on a basis of decreased labor cost. This would depend upon the number of analyses which were run daily and the fact that wet methods would still be necessary for the determination of calcium oxide and silicon dioxide, regardless of subsequent improvements in spectrographic methods.

We are inclined to doubt the value of the authors' semiquantitative method for cement or cement raw materials. It is hard to see where analyses "differing not more than 30 to 50 per cent from the true values" would be particularly useful, even for survey work. Obviously the method does not apply to major constituents, and even the minor constituents which are important certainly need to be determined with greater accuracy. Today there is more interest in alkalies than in any other minor constituents in cement, but the authors specifically mention alkalies as being among the least satisfactory determinations by this method.

Mr. M. F. Hasler (*author's closure by letter*).—In reply to the question of W. J. McCoy, one of the important features of the spectrographic method is that it is not necessary to put the sample in solution. Therefore, in cases of raw material analysis just as in cement analysis, the spectrographic method can be applied directly.

The simple d-c. arc method for sodium and potassium determinations is quite rapid. It requires the weighing and mixing of nine parts sample with one part lithium carbonate. Two 10-mg. portions of the mixture are than weighed and loaded into electrodes. These are arced 10 sec. each to produce duplicate spectrograms of the sample. This can easily be done in 5 min. for a single sample. It then takes 6 min. to develop, wash, and dry the film and a few minutes more to read and calculate the sodium and potassium values in duplicate. Thus, in from 15 to 20 min., both these determinations can be made in any type of material that has been previously standardized.

If a large number of samples were to be analyzed, this time could be reduced to about three man minutes per alkali determination, as up to seventeen samples can be analyzed on a single strip of film in the spectrograph. This then spreads the film developing time, etc., over a large number of analyses.

This would thus indicate that the advantages of the spectrographic method, for the determination of alkalies alone, are real in the case of samples not readily soluble in acid.

Concerning the statements of W. C. Hanna, these are essentially correct. By way of comment, we should like to take this opportunity to provide times of analysis by the various methods that we feel are particularly suited to the quantitative analysis of cement and its raw materials.

The first of these are the d-c. arc methods described under the headings, "Method for Refractory Elements Without Fluxing" and "Method for Alkalies." Starting with a well-ground sample, it requires about 10 min. to finely grind a small portion, make the necessary weighings in duplicate for both methods, and load the electrodes. Placing the samples in the arc-spark stand and arcing as required takes another 6 min. for duplicate samples. Thus, for eight samples, this would require just about two man hours. Developing the film, reading the transmissions of seven spectrum lines per spectrogram, and calculating the percentage composition would require another man hour. Thus, one technician could analyze eight samples in duplicate by this method in about 3 hr.

The second of these is the interrupted d-c. arc method described under the heading, "Method of analysis with briquets and with fluxing prior to discharge." In this method, it takes about 15 min. total handling time to prepare duplicate briquets of a sample. This in cludes the time before fusion and after, but not the fusing time. Thus, about 2 hr. would be consumed in preparing eight duplicate samples, plus 1 hr. for fusion. Arcing would require another half hour and developing, reading and calculating another hour. Thus, three and one half man hours per eight samples in duplicate are required exclusive of the fusion time. However, since this fusion does not require the attendance of a man, it can usually be fitted into a routine without much loss of time.

EFFECT OF DELAYED MIXING OF PREBATCHED MOIST AGGREGATES AND CEMENT ON THE STRENGTH AND DURABILITY OF CONCRETE*

By W. H. Price[1] and J. W. Robison[1]

Synopsis

Cement and moist aggregate have been batched and hauled long distances before mixing, and where such procedures are employed the cement may remain in contact with the damp aggregates for hours before the ingredients are mixed and placed in the structure. Tests made to determine the effect of this delay on the properties of the concrete indicate that for a moisture content of 7.5 per cent in the sand and 1.5 per cent in the gravel and with a constant slump, the compressive strength of the concrete is reduced by 4.5 per cent for each hour of delay between batching and mixing. The durability of the concrete, as measured by freezing-and-thawing tests, is improved as the interval between batching and mixing is increased up to about two hours.

The tests described in this paper were made to determine the effect of prebatching of cement and moist aggregate on the properties of the concrete lining of the 13-mile long Alva B. Adams tunnel. However, the results obtained should be applicable to other jobs where there is a delay between the batching and mixing of the concrete ingredients.

Since there was insufficient room in the 11.75-ft. diameter tunnel for batching of the concrete ingredients, they were batched outside the tunnel and hauled in small railroad cars to a point near the lining work, where they were dumped onto a belt which conveyed them to a hopper from which they could be conveyed to the concrete mixer. It was indicated that considerable time might elapse between the batching of the cement and moist aggregate and the mixing of the concrete. In order that the effect of this delay on the properties of the concrete could be determined, a series of mixes was made with intervals between batching and mixing ranging up to 3 hr. The aggregate was carefully prepared to represent the average moisture condition expected to be encountered in the tunnel construction, and a small batching arrangement simulating that proposed for use in the tunnel was used to provide a representative mixing of the aggregate and cement in the batching process. Figures 1 and 2 show the batching hopper and conveyor used in the laboratory tests. This equipment was previously used for studying bin designs and batching sequences.

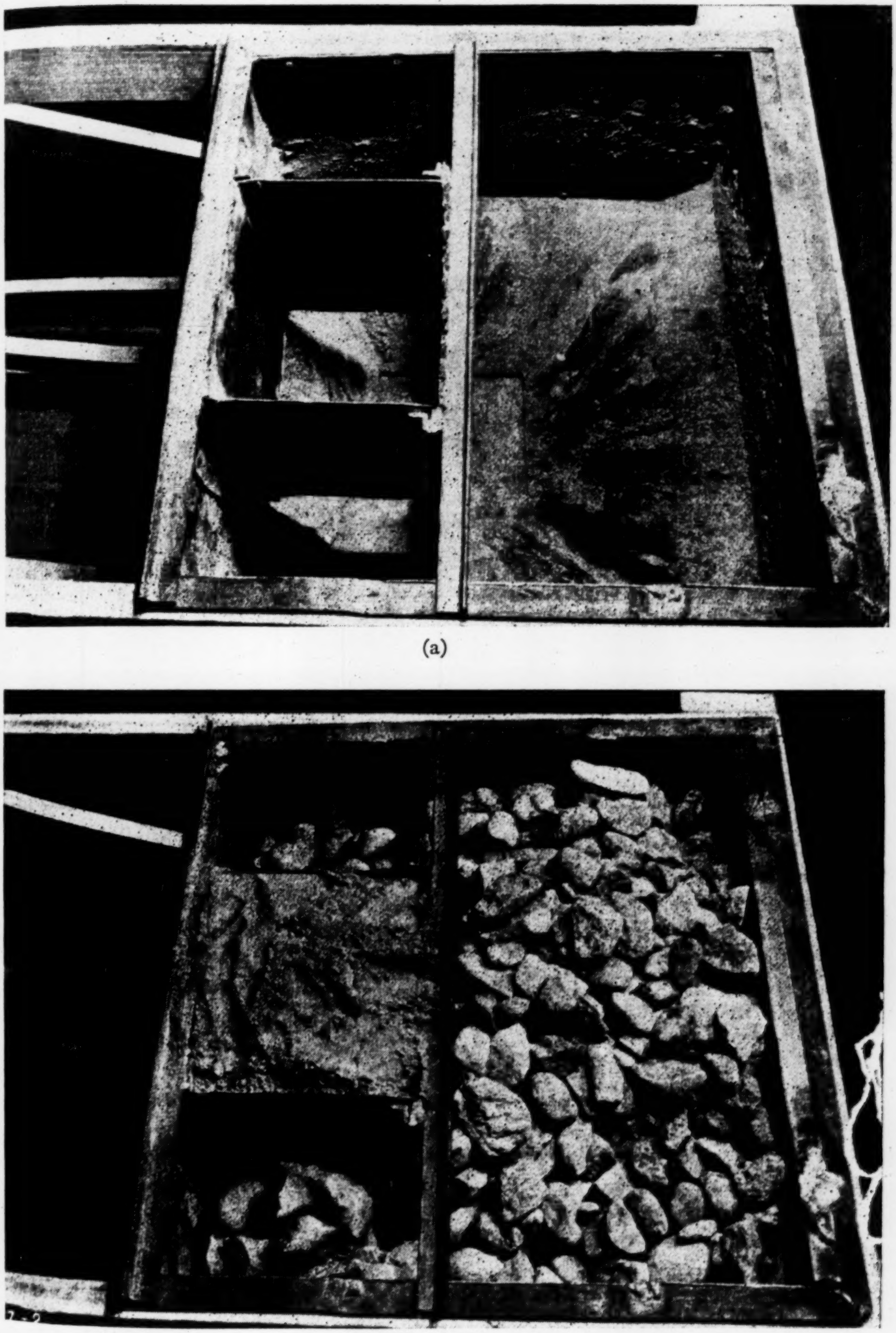
Description of Tests

Comparative tests were made on the fresh and hardened concrete for intervals of 0, 30, 60, 90, 120, 150, and 180 min. between batching and mixing of the concrete. Three sets of seven batches each

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Materials Engineers, U. S. Bureau of Reclamation, Denver, Colo.



(a)

(b)

FIG. 1.—(a) Top View of Model Tunnel-Car Hopper Showing Relative Position of Cement and Aggregate Compartments. (b) View of Model Tunnel-Car Hopper Loaded with Cement and Aggregate.

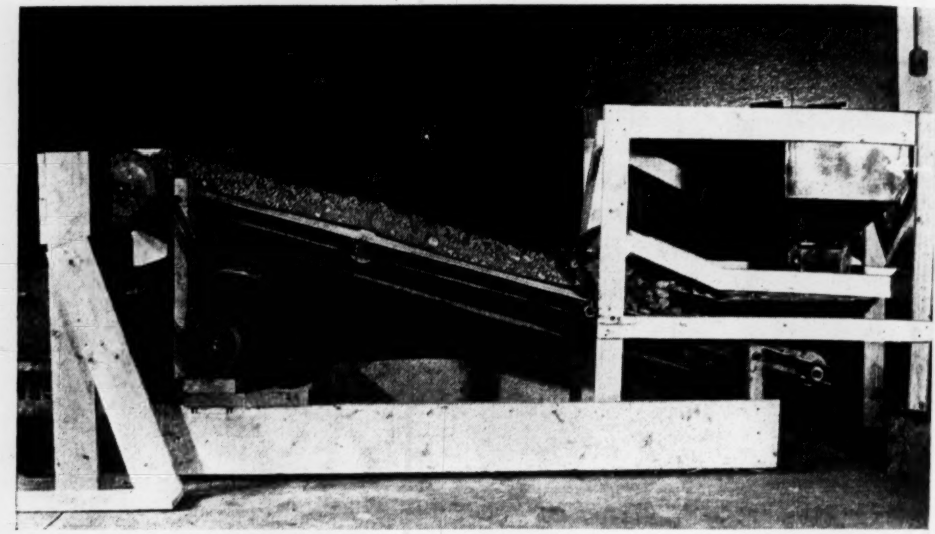


FIG. 2.—General View Showing Concrete Batch Being Discharged over Belt Conveyor from Model Tunnel-Car Hopper Shown in Fig. 1 into Mixer Storage Hopper. (Arrow indicates model tunnel-car hopper.)

TABLE I.—MIX DATA AND STRENGTH RESULTS.

	Batch Number	Delay Between Batching and Mixing, min.	Net Water-Cement Ratio, by weight	Slump, in., Avg. of 2 Specimens	Powers Remolding, Number of Jigs	Compressive Strength, psi.		Elastic Properties 28-day cyl.		Workability			Notes
						7 days	28 days (avg. of 2)	$E \times 10^{-6}$	r	Rodability	Finish	Segregation	
Set No. 1 Uncovered	1	0	0.50	2	47	3 710	5 395	3.42	0.16	Good	Good	None	Mix slightly harsh
	2	30	0.53	4½	72	3 130	4 730	3.41	0.14	Good	Good	None	Mix slightly harsh
	3	60	0.52	3	70	3 450	5 065	3.47	0.18	Good	Good	None	Mix slightly harsh
	4	90	0.52	3	50	2 790	4 535	3.35	0.15	Good	Good	None	Mix slightly harsh
	5	120	0.53	3	56	3 150	4 785	3.61	0.17	Good	Good	None	Mix slightly harsh
	6	150	0.54	3	56	2 600	4 290	3.43	0.16	Good	Good	None	Mix slightly harsh
	7	180	0.53	2¾	68	2 900	4 340	3.38	0.16	Good	Good	None	Mix slightly harsh
Set No. 2 Fog room	8	0	0.50	2¾	56	3 400	4 980	3.49	0.14	Good	Excellent	None	Good concrete
	9	30	0.51	3	47	3 330	4 710	3.31	0.19	Good	Excellent	None	Good concrete
	10	60	0.52	3½	43	3 110	4 635	3.49	0.14	Good	Excellent	Slight	Good concrete
	11	90	0.51	2¾	70	3 170	4 555	4.63	0.21	Good	Excellent	Slight	Crumbly
	12	120	0.52	2¾	64	3 330	4 795	3.45	0.16	Good	Good	Slight	Crumbly
	13	150	0.53	3	49	3 010	4 450	3.44	0.18	Good	Good	Slight	Crumbly
	14	180	0.57	4¼	39	2 370	3 730	3.30	0.16	Good	Good	Slight	Crumbly
Set No. 3 Covered	15	0	0.51	2¾	62	3 860	5 405	2.93	0.18	Good	Excellent	None	Good Concrete
	16	30	0.50	3	64	3 560	4 975	3.27	0.15	Good	Excellent	None	Good Concrete
	17	60	0.53	3½	76	3 770	4 625	3.31	0.18	Good	Excellent	None	Good Concrete
	18	90	0.53	3	37	3 080	4 385	3.13	0.22	Good	Excellent	None	Good Concrete
	19	120	0.53	3	36	3 430	4 765	3.35	0.18	Good	Good	None	Slightly harsh
	20	150	0.55	3½	43	2 760	4 230	3.00	0.17	Good	Good	None	Slightly harsh
	21	180	0.54	3½	46	2 830	4 405	3.21	0.17	Good	Good	None	Slightly harsh

Temperature of fresh concrete after mixing varied from 76 to 78 F.
Cement content varied from 1.44 to 1.42 bbl. per cu. yd.
Unit weight of fresh concrete varied from 150.7 lb. per cu. ft. for the lower water cement ratios to 149.0 lb. per cu. ft. for the higher water cement ratios.
Mix parts by weight: 1:2.30:3.70 per cent sand = 38.
Gravel grading: No. 4 to ⅜ in., 25 per cent; ⅜ to ¾ in., 30 per cent; ¾ to 1½ in., 45 per cent.
Sand Grading: pan, 6 per cent; No. 100, 13 per cent; No. 50, 21 per cent; No. 30, 30 per cent; No. 16, 19 per cent; No. 8, 10 per cent; No. 4, 1 per cent.
Fineness modulus = 2.77.

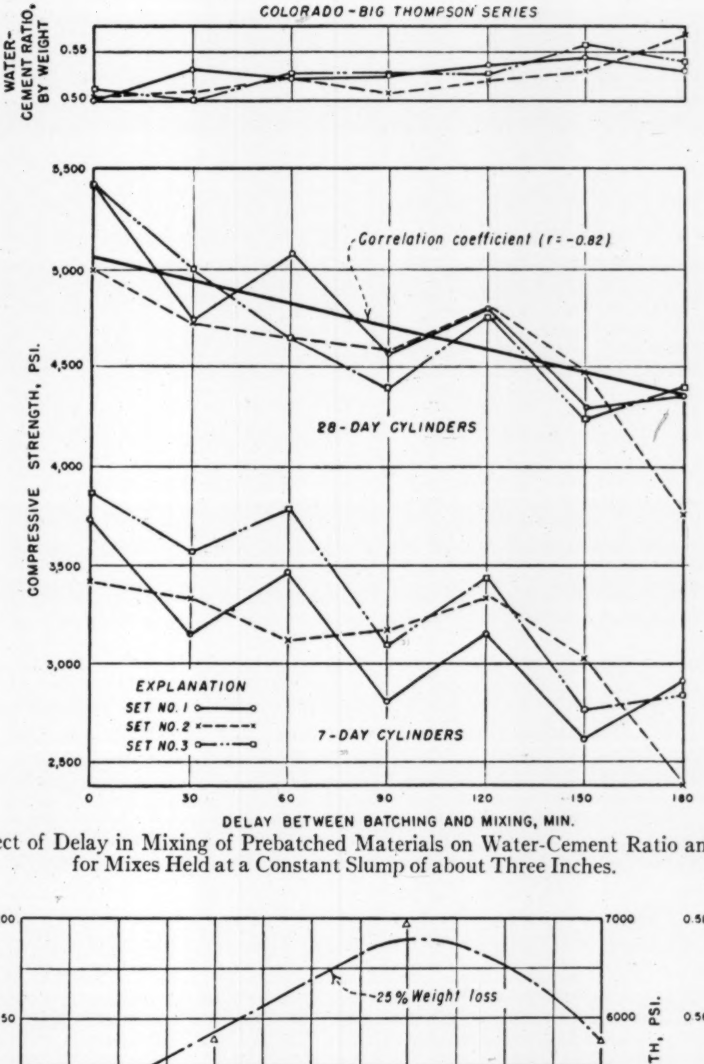


FIG. 3.—Effect of Delay in Mixing of Prebatched Materials on Water-Cement Ratio and Strength for Mixes Held at a Constant Slump of about Three Inches.

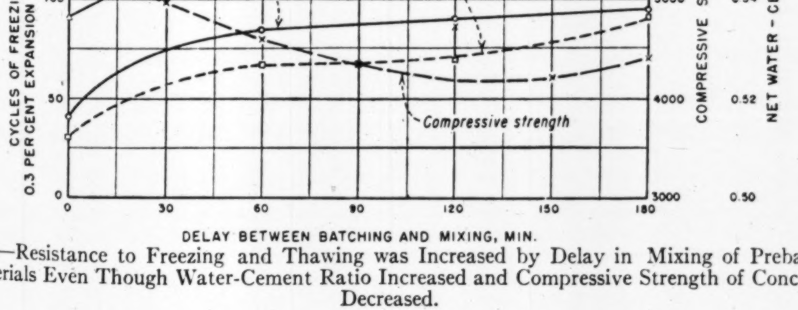


FIG. 4.—Resistance to Freezing and Thawing was Increased by Delay in Mixing of Prebatched Materials Even Though Water-Cement Ratio Increased and Compressive Strength of Concrete Decreased.

were made in this series of tests. The first set of seven mixes was allowed to remain uncovered in the laboratory between the time of batching and mixing. The second set was placed uncovered in a 100 per cent relative humidity room until time of mixing, and the third set was covered with a damp cloth until mixed. The results of the tests are shown in Table I and Figs. 3 and 4.

To assure uniform moisture conditions for the tests, the coarse aggregate was soaked in water for 24 hr., after which each size of gravel was placed on a screen having the next smaller size opening and allowed to drain for 15 min. before using. After draining and prior to batching, moisture determinations were made on each gravel size. Averages of these determinations are as follows: No. 4 to $\frac{3}{8}$-in., 2.1 per cent; $\frac{3}{8}$ to $\frac{3}{4}$-in., 1.3 per cent; and $\frac{3}{4}$ to $1\frac{1}{2}$-in., 0.4 per cent.

The sand used in the tests was dried and separated into individual sizes, after which it was recombined to give the grading shown at the bottom of Table I. Water equal to 8 per cent of the dry sand weight was added, and sand and water were mixed together. After mixing, the sand contained 7.5 per cent free moisture.

A Type II cement which had no premature stiffening tendencies was used in the tests. This cement had an initial time of set of 2 hr. and 20 min. The temperature of the mixing water was maintained at 70 F.

The ingredients for each batch were weighed to the nearest 0.01 lb. before they were placed in the model tunnel-car hopper, in the following order: fine gravel, sand, and coarse gravel. The cement was then placed in a separate compartment. As soon as the batch was ready it was discharged over the belt conveyor into the concrete hopper, as shown in Fig. 2. Each set of seven mixes was made on separate days, and all batching and mixing for one set was performed during a period of approximately six hours.

All batches were mixed in a 2-cu. ft. tilting mixer. To maintain a uniform condition from batch to batch, the mixer was washed thoroughly and allowed to drain for two minutes prior to charging. Each batch was mixed for five minutes, and corresponding to field practice, sufficient water was added to each mix to maintain a slump of about 3 in. After each batch was mixed, it was discharged into a wheelbarrow and thoroughly mixed with a shovel to eliminate segregation occurring during the dumping of the mixer. After this remixing, two slump tests and a remolding test were made, and the unit weight and concrete temperature were determined before casting specimens.

Three 6 by 12-in. cylinders were cast from each batch for compressive strength and elastic properties determinations. The molds were filled in two lifts, and each lift was vibrated by an internal vibrator which was inserted into the layer at each of three locations for a period of ten seconds each. Three 3- by 6-in. cylinders were cast from the 0, 60, 120, and 180 minute batches of the cloth-covered third set for freezing-and-thawing durability tests. Concrete for durability specimens was wet-screened to remove the $\frac{3}{4}$ to $1\frac{1}{2}$-in. aggregate before filling the molds in two layers by hand rodding each layer 25 times with a $\frac{3}{8}$-in. diameter rod. Each end of the 3- by 6-in. cylinders was fitted with a stainless steel insert for length measurements. Immediately after the molds were filled, they were placed in the 70 F. fog room. The molds were stripped from all specimens after 24 hr. and the specimens were rotated in position each day for the first seven days of their fog-curing period to eliminate variations in curing during this early strength developing period.

Bleeding tests were made in accordance with Designation 25 of the Reclamation Concrete Manual on the 30, 90, and 150 min. batches of set No. 3.

One specimen from each batch was tested in compression at the age of seven days. The remaining two specimens from each batch were tested in compression at the age of 28 days. Young's modulus and Poisson's ratio determinations were made on one of the 28-day specimens. The results of these tests are presented in Table I and Fig. 3.

The 3 by 6-in. durability specimens were fog cured for 28 days after which they were removed to the 70 F., 50 per cent relative humidity room where they remained for 152 days before being subjected to the freezing-and-thawing tests. They were weighed and measured periodically during drying to determine comparative shrinkage and moisture losses. After the 152 days of drying and prior to starting the cycles of freezing and thawing, the specimens were soaked in water for 96 hr. The three durability cylinders were placed on end in close-fitting cylindrical rubber containers, covered with water, and then lowered into the freezing cabinet where the containers were surrounded with brine at a temperature of +5 F. A cycle consisted of $1\frac{1}{2}$ hr. of freezing at +5 F. and $1\frac{1}{2}$ hr. of thawing in water maintained at 70 F. The cylinders were weighed and measured for expansion each week after every ten cycles. Failure was assumed at 25 per cent weight loss. The results of the freezing-and-thawing tests are shown in Fig. 4.

DISCUSSION OF TEST RESULTS

Figure 3 shows that it was necessary to increase the water-cement ratio from about 0.50 to 0.55 to maintain the slump as the interval between batching and mixing was extended to 3 hr. Although the slump was maintained nearly constant at 3 in., the workability of the concrete was definitely reduced after a delay of two hours and the concrete was slightly crumbly after this period. This reduction in workability was not reflected by the number of jigs required for remolding in the Powers' apparatus. Figure 3 also shows the decrease in compressive strength resulting from the delay in mixing of batched moist aggregate and cement. The straight line on this figure is the line of best fit for the three sets of tests. This line of regression shows a decrease in compressive strength of 231 ± 37 psi. per hour of delay. Statistically, this value is considered to be highly significant in view of the indicated reliability of the test results as shown by a correlation coefficient of -0.82.

Figure 4 indicates that the durability of the concrete as measured by the freezing-and-thawing tests is improved as the delay in mixing batched moist aggregate and cement is extended up to about two hours, despite the increase in water-cement ratio and decrease in the compressive strength. Other tests made in this laboratory have shown that the durability of concrete increases as the age of the cement stored in closed drums increases up to about eight months. Apparently some seasoning of the cement is beneficial from the durability standpoint.

Other measurements made in this series of tests showed no significant differences in the drying shrinkage, moisture loss or absorption of the hardened concrete. The bleeding of the fresh concrete was decreased slightly by the delay in mixing.

DISCUSSION

MR. C. E. PROUDLEY[1] (*presented in written form*).—The North Carolina State Highway Laboratory does not have information to compare directly with that secured by Messrs. Price and Robison, but the same objective was in mind when a study was undertaken about six years ago to determine the effect of delay between batching and mixing of paving concrete on its flexural strength. The desire for this information was created by the occurrence on paving jobs of breakdowns in equipment which necessitated holding loaded batch trucks for periods varying from several hours up to the following day. The contractor wished to salvage what he could from the batches standing in the trucks, and it became our practice, based on lack of information, to require that he add 50 per cent cement to the batch.

Tests made in the laboratory simulated the dimensions of batch trucks, moisture conditions usually prevailing in the aggregates, and the average proportions for pavement concrete. In those days very few bulk cement plants were used on paving jobs in North Carolina, and the usual size of batch was 6-bag. It seemed reasonable, therefore, to determine the effect of adding 1, 2, and 3 bags to the batch.

The batch boxes were $11\frac{5}{8}$ by $11\frac{5}{8}$ by 24 in., which were roughly proportional dimensions to those for 6-bag batches used in the field. Canvas covers for the boxes were used as required by specifications. The laboratory temperature was 70 to 80 F. during batching and subsequent mixing and molding. The coarse aggregate was crushed granite graded uniformly from 2-in. to No. 4. The fine aggregate was a washed, silica sand, having a fineness modulus of 2.45. The proportions by weight were 94:213:363 with sufficient water to give a 2-in. slump. Preparation of the batch, mixing and molding operations were repeated on different days to minimize incidental variations. Each set of specimens comprised three beams on which two flexure tests were obtained, making six breaks for each condition.

The materials were arranged in the batch box with dry stone in the bottom, sand containing 5 per cent moisture in the middle and the portland cement on top, covered with a canvas. The boxes were allowed to stand for periods of 4, 8, 12, and 16 hr. Other batches were mixed immediately to give the conditions approximating a normal concrete paving job. Cement was added to batches in amounts of $16\frac{2}{3}$, $33\frac{1}{3}$, and 50 per cent, representing the 1, 2, and 3-bag additions to 6-bag batches.

The accompanying Table II gives the data concerning the variations in water content required to give a 2-in. slump as the periods of delay increased from 0 to 16 hr. and, also, with increase in cement content. The modulus of rupture included in the tabulation was determined at 14 days with third-point loading. From this information it may be computed that approximately 0.27 per cent

---

[1] Chief Materials and Test Engineer, State Highway and Public Works Commission, Raleigh, N. C.

TABLE II.—FLEXURAL STRENGTH OF CONCRETE MIXED AT VARIOUS INTERVALS AFTER BATCHING, WITH AND WITHOUT ADDITIONAL CEMENT.

Specimens 8 by 6 by 30-in. beams tested in accordance with A.S.T.M. specification C 78 – 38.

All specimens cured with wet cotton mats during first 24 hr. and in standard damp room to age of 14 days. Each value is the average for 3 beams, 2 breaks on each beam.

Materials were type I portland cement, washed sand from Goldsboro and crushed granite from Greystone. Proportions by weight: 94:213:363.

No moisture in coarse aggregate; 5 per cent moisture in fine aggregate.

Set	Delay in Mixing Time, hr.	Cement Added to Batch After Delay, per cent by weight	Average Water Content, gal. per bag	Slump, in.	Modulus of Rupture at 14 days, psi.
No. 1	0	0	6.2	2.0	581
2	4	0	6.2	1.9	574
3	4	16–2/3	5.6	1.9	657
4	4	33–1/4	5.3	2.0	666
5	4	50	5.0	2.0	712
6	0	0	6.2	2.0	581
7	8	0	6.2	1.8	567
8	8	16–2/3	5.7	1.9	635
9	8	33–1/3	5.4	2.2	682
10	8	50	5.0	2.0	714
11	0	0	6.2	2.0	581
12	12	0	6.3	1.9	563
13	12	16–2/3	5.8	2.0	635
14	12	33–1/3	5.6	2.3	638
15	12	50	5.1	2.2	693
16	0	0	6.2	1.7	581
17	16	0	6.5	2.0	559
18	16	16–2/3	5.9	2.0	616
19	16	33–1/3	5.5	2.0	668
20	16	50	5.2	2.1	692

strength was lost for each hour delay in mixing. Also increasing the quantity of cement resulted in an increase in flexural strength at 14-days of between 0.5 and 0.75 per cent for each 1 per cent increase in cement. An approximate figure to be used for the addition of cement to offset the effect of each hour of delay in mixing would be one-third of one per cent, or 1 per cent for each 3-hr. delay.

This is a very much lower figure than was shown for compressive strength by Price and Robison; however, the test conditions are not comparable nor can it be assumed that the materials involved would behave in the same manner when mixed in concrete. This discussion, therefore, only adds to the information available and gives something pertaining to flexural strength which was not included in the paper under discussion.

It may be of interest to note that in North Carolina we instruct concrete pavement inspectors to require that not less than 10 per cent additional cement be added to delayed batches which, obviously, is sufficient to take care of the average condition. Should the batch be carried over until the next day we still require the addition of 50 per cent cement since there are many conditions which could occur on the job which were not covered by our study, such as higher moisture content of the aggregates, some blending of the cement and aggregates caused by hauling, leaky canvas covers through which rain or dew might affect the cement, and similar conditions which would add to the partial hydration of the cement during the delay.

MR. F. O. ANDEREGG.[2]—Having occasion to check up on a mortar intended for painting stone work, a batch of cement and sand in about a 1:3 mix was prepared in a small covered mixer. Specimens were made up for compression testing and volume change measurement immediately after mixing, after standing 2 and 4 hr. covered and followed by remixing, but without adding water, and at 4 hr. with water added to bring back to the original consistency of about 40 per cent flow. A similar batch was made with an admixture.

The shrinkage measured after one day in the damp closet and six days exposed to the laboratory air was least with the portland-cement mortar made up immediately and was greatest after retempering at 4 hr. The compressive strengths, as well as ability to effect good contact with stone units, appeared to be best on the 2-hr. specimens. At 4 hr. both these properties were markedly diminished, especially when the admixture was used.

---

[2] Director, Materials Research, John B. Pierce Foundation, Raritan, N. J.

The volume changes observed were different from those made under similar circumstances 20 yr. ago, when standing up to about 2 to 4 hr. seemed to reduce shrinkage. Modern cements are more finely ground than then, resulting in certain changes in properties. A complete report of these tests will be submitted to A.S.T.M. Committee C-12 on Mortar for Unit Masonry.

A paper presented 20 yr. ago by H. F. Gonnerman and published in the November 1928 Report of the Director of Research of the Portland Cement Association describes experiments showing the "small loss in compressive strength due to standing for periods up to 6 hr. (protected from evaporation) in concrete remixed without the addition of water." It would be very interesting to repeat some of these experiments and compare the results with the Price and Robison experience reported in 1948.

MR. F. H. JACKSON.[3]—I take it, that Mr. Anderegg refers entirely to drying shrinkage and not settlement shrinkage.

MR, ANDEREGG.—Yes, that is right.

MR. JACKSON.—I should like to ask the authors whether they reached any conclusions as to why the concrete exhibited greater durability as the period of delayed mixing was increased. That was rather surprising and interesting.

MR. WALTER H. PRICE (*author*).—Tests which we now have in progress may give us an answer to this increased durability with time of delay. These tests will be discussed in our closing remarks.

MR. M. A. SWAYZE.[4]—I should like to ask one question, and then, contingent on the answer, I might have some discussion.

As I understand it, the cement was a type II cement, thoroughly premixed with damp aggregates?

MR. PRICE.—That is right.

MR. SWAYZE.—Here is the point of my questions. There may be some dangerous conclusions drawn from this paper. I want to relate an experience of 18 yr. ago, and another one of this past spring, where a user ordered a batch of ready-mixed concrete, dry-mixed. In other words, they simply added the cement dry to the sand and gravel, both of which had surface moisture in normal amounts. In the case 18 yr. ago they delivered the batch to a job where they had some tide-water construction that could not go in until about 11:00 o'clock at night. They allowed this batch to stand for about 5 to 6 hr. on a hand mixing platform. Then the workmen added water, turned it over with shovels and placed it into the forms.

The next morning it did not look too good, but they gave it an additional 24 hr. At the end of 36 hr. very much to their surprise, they found that the whole mass had no more cohesion than the original sand and gravel would have had. In other words, the cement during that 5 or 6-hr. waiting time had stuck to the grains and had hydrated enough so that there was no further cementing action after adding water and remixing.

The same thing recurred again this spring.

Again, a batch of type I cement was premixed with damp aggregate containing just enough water to dampen the cement, then allowed to stand for 4 or 5 hr. That cement also hardened sufficiently so that it would take no more water. It was not ground loose from the aggregate and redistributed on remixing, and the job was a complete failure.

So there should be some warning in connection with the authors' results. Their conditions may have been different.

---

[3] Principal Engineer of Tests, Public Roads Administration, Federal Works Agency, Washington, D. C.

[4] Director of Research, Lone Star Cement Corp., New York, N. Y.

MR. PRICE.—The results given in the paper are for delays up to 3 hr. Since the paper was written, however, we have made additional tests with periods of delay between batching and mixing extending up to 24 hr. These results showed that with the particular cement used which had an initial set of 6 hr. and final set of 9 hr., a concrete having considerable strength could be obtained after 12 hr. delay. Lumps began to form in the mixture after 6 hr.; after 12 hr. these lumps had to be broken up before charging the mixer. In all cases the lumps were completely broken up during the 5-min. mixing period. For concrete having a faster setting this might be different, and for that reason the Bureau of Reclamation requires that the batch be wasted after 6 hr. of contact of the cement and aggregate. The specifications currently used by the Bureau are as follows:

> When bulk cement and aggregates are hauled from a central batching plant to the mixers the cement for each batch shall
>
> (1) be placed in an individual compartment which, during transit, will prevent the cement from intermingling with the aggregates and will prevent loss of cement, or shall
>
> (2) be completely enfolded in and covered by the aggregates by loading the cement and aggregates for each batch simultaneously into the batch compartment. Each batch compartment shall be of sufficient capacity to prevent loss in transit and to prevent spilling and intermingling of batches as compartments are being emptied. If the cement is enfolded in aggregates containing moisture, and delays occur between filling and emptying the compartments, the contractor shall, at his own expense, add extra cement to each batch in accordance with the following schedule:

Hours of Contact Between Cement and Wet Aggregate	Additional Cement Required, per cent
0 to 2	0
2 to 3	5
3 to 4	10
4 to 5	15
5 to 6	20
Over 6	Batch will be rejected

MR. W. C. HANNA.[5]—I had an interesting experience that does not compare exactly with that of either the authors or Mr. Swayze.

I refer to a case where I happened to be on a job, when concrete on a main beam over a band stand was being poured, a very expensive band stand where they had waste molds. The contractor had a mixer on the job and he had the beam about half poured. It was very necessary that there should be no pouring joint or cold joint across the beam but in the middle of the pour the concrete elevator broke down. I happened to be there at this time. The contractor turned to me and said, "I'm ruined!" He thought he never could salvage the job.

I told him that since he had plenty of men available he should just keep the gang tamping. He thought it would do no good, since he could not get the elevator for 4 or 5 hr.

But he kept the gang of men working there for, as I recall, 6 hr., tamping back and forth, and then went ahead with the job, and put the new concrete on top of the old. It was a fine job and has been in for 25 yr.

MR. H. F. GONNERMAN.[6]—I should like to ask the authors whether any air content determinations were made to see if the air content might have had an influence on the durability of the concrete?

In tests we have been making recently with non-air-entraining cements we find a good correlation between the volumetric ratio of the air content to the freezable water content of the concrete, and the resistance of the concrete to freezing and thawing. Therefore if the air content of the concrete were known one might find the reason for the increase in durability.

---

[5] Chief Chemist, California Portland Cement Co., Colton, Calif.

[6] Director of Research, Research Laboratory, Portland Cement Association, Chicago, Ill.

MR. PRICE.—In the series of tests reported in the paper, we did not make air content determinations. The unit weight of the concrete varied as listed at the bottom of Table I, that is, from 150.7 to 149.0 lb. per cu. ft. The air content of the concrete could not be estimated accurately from the unit weights because of the change in the specific gravity of the cement caused by its partial hydration.

We are aware of the effect that a small amount of air can have on the resistance of concrete to freezing and thawing. Recently we tested the same aggregate with two different brands of cements having nearly the same composition and fineness. Very poor resistance to freezing and thawing was obtained with one brand and good durability with the other. An analysis of the results showed that the cement which produced the poor durability entrained practically no air—less than 0.2 per cent—whereas about 1 per cent air was entrained with the other cement.

MR. W. E. PARKER[7] (*by letter*).—In 1941 the Hydro-Electric Power Commission Laboratories undertook a study of the effect of delayed mixing for the Subcommittee on Ready-Mixed Concrete of A.S.T.M. Committee C-9, the object being to provide some guidance in the matter of permissible delays between batching and mixing. While the test conditions differed in certain respects from those described by the authors, it is felt that the results are relevant to the general problem of delay after intimate contact of cement and aggregate. Thorough initial mixing was intentionally provided in these tests in order to equal the severest condition that might occur in transit mixers.

The program, which consisted of six similar series, was intended primarily to study the effect of delays between batching and mixing and to cover the secondary factors of mix proportions and moisture content of the aggregates. Within each series the only variable was the time of delay which ranged in $\frac{1}{2}$-hr. increments from zero to $1\frac{1}{2}$ hr. with additional test points for 3 and 5 hr. Each test condition was represented by a single batch from which three standard compression cylinders were molded for tests at 28 days. The six series included mixes with cement contents of about 450 , 540, and 620 lb. per cu. yd. and with the free moisture in the sand ranging from 3 to 6 per cent.

The materials used throughout the tests were type I cement from a single mill; sand from the mouth of the Niagara River, batched in the moist condition; and crushed limestone graded from No. 4 to $1\frac{1}{2}$ in., batched dry. Absorption of the stone was 0.5 per cent, by weight.

The solid ingredients of a batch were combined initially by hand mixing, then stored in covered pans for the specified period of "contact" before being transferred to a Lancaster mixer. As mixing began, water was added to produce water-cement ratios of 0.75, 0.61, and 0.52 by weight, corresponding to the three cement contents stated previously. In the control mixes, where no delay occurred between batching and mixing, the slump ranged from 4 to 7 in.

Although all of the observed properties of the concrete showed progressive changes corresponding roughly to the length of delay, the test results were too scattered to permit an evaluation of the importance of moisture content of the sand or of changes in mix proportions.

---

[7] Assistant Testing Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Ontario, Canada.

In addition, the scale of the tests was insufficient to assess the significance of slight departures from straight-line relationships. But since such trends were apparent in the average curves representing the six series of tests, the general shapes of the curves will be indicated.

The average results for 28-day compressive strength indicated a progressive decrease with time of delay, the reductions for 1½ and 5-hr. periods being about 8 and 18 per cent. Flow and slump appeared essentially unaffected for periods up to 1½ hr., and thereafter decreased in a fairly uniform manner to respective 5-hr. values of 88 and 63 per cent. Unit weight, on the other hand, was lowered about ½ per cent as the result of ½-hr. delay, while the total decrease in the 5-hr. period averaged 1½ per cent. A few tests for air content by the pycnometer method indicated a gradual increase of 0.9 percentage point for 3 hr. delay and 1¼ points for 5 hr.

The above results appear to provide general corroboration of the authors' findings. The slightly greater strength reduction shown by their tests is probably accounted for by the higher moisture content of the aggregates. The results also suggest that the observed properties continue to change progressively for periods of delayed mixing amounting to as much as 5 hr.

TABLE III.—EFFECT OF DELAYED MIXING OF PREBATCHED MOIST AGGREGATES AND CEMENT ON THE STRENGTH AND DURABILITY OF CONCRETE.

NOTES.—Average slump for the concrete was 3 in. The average concrete temperature was 68 F. Aggregate grading similar to values reported in Table I of the original article.

Delay, hr.	Net Water-Cement Ratio, by weight	Compressive Strength, 6- by 12-in. Cylinders, psi.		Weight of Fresh Concrete, lb. per cu. ft.	Added Cement, per cent	Air Content, per cent[a]	Bleeding, per cent
		7 days	28 days				
Series A							
0	0.51	3180	4990	149.6	0	...	...
1	0.49	2850	4690	149.0	5	...	...
2	0.48	3240	5050	149.6	10	...	...
3	0.47	3180	5040	149.0	15	...	...
4	0.46	3310	5290	149.0	20	...	...
5	0.46	3570	5540	149.2	25	...	...
6	0.45	3630	5780	149.0	30	...	...
Series B							
0	0.51	3030	5020	149.0	None	...	5.8
1	0.52	2460	4540	149.4	"	...	5.6
2	0.52	2480	4240	148.8	"	...	4.8
3	0.54	2300	4020	148.8	"	...	5.4
4	0.56	2140	3740	148.4	"	...	4.4
5	0.56	2250	3860	149.2	"	...	3.7
6	0.56	1810	3490	148.4	"	...	4.5
8	0.57	1900	3840	148.6	"	...	4.5
10	0.58	1650	3300	147.2	"	...	3.6
12	0.62	1230	2670	146.0	"	...	5.9
16	0.69	530	1260	142.1	"	...	...
20	0.77	270	720	140.3	"	...	...
24	0.83	40±	410	137.1	"	...	14.0
Series C							
0	0.51	3310	5570	148.5	0	1.5	2.0
1	0.51	3150	4980	148.2	0.5	1.6	...
2	0.51	3340	4900	148.2	0.9	1.7	2.0
3	0.51	3180	5260	147.7	1.5	1.8	...
4	0.51	3010	4870	147.3	1.9	1.9	1.7
5	0.51	2910	4800	146.8	2.4	1.9	...
6	0.51	2980	4920	148.0	2.4	1.7	2.0
7	0.51	2910	4990	147.6	2.9	1.9	...
8	0.51	2870	4730	147.6	3.7	1.9	2.0
9	0.51	2720	4680	146.5	7.2	2.1	...
10	0.51	2560	4590	146.4	10.8	2.0	2.5
11	0.51	2760	4830	145.7	15.7	2.1	...
12	0.51	2530	4430	145.1	23.1	2.2	...
13	0.51	2280	3680	143.4	30.1	2.7	...
14	0.51	2160	4190	144.1	43.5	2.0	3.4
15	0.51	2210	4260	143.6	44.7	1.8	...
16	0.51	2060	4190	143.9	44.7	2.1	3.2
18	0.51	1930	3850	142.5	57.5	2.2	...
20	0.51	1970	3840	142.5	70.0	2.1	4.2
24	0.51	1870	3510	140.1	80.7	3.2	5.0

[a] Air content obtained by air meter on series C only.

MESSRS. PRICE AND J. W. ROBISON (*authors' closure*).—Since the original paper was written, three additional series of tests were made to investigate the effects of delayed mixing of batched moist aggregates and cement on the properties of the resulting concrete. In all tests, the aggregate was the same as that described in the original paper with the moisture content about 1.8 per cent in the gravel and 7.5 per cent in the sand. The type II cement used in the later tests had an initial set of 6 hr. and a final set of 9 hr. , whereas the cement used in the tests described in the original paper had an initial set of 2 hr. and 20 min. and a final set of 4 hr. It is believed that the time of set of the cement is very important in the results obtained from batches delayed more than 3 hr.

The aggregate and cement were thoroughly mixed with a shovel in every case, because Bureau specifications require that the cement be completely enfolded in and covered by the aggregate to prevent it from being blown away during transit to the mixer. All concrete of the later series had lower resistance to freezing and thawing than did the concrete of the original tests. This difference is probably due to the change in cement and in the method of curing prior to testing. In the original tests the specimens were fog cured at 70 F. for 28 days, then stored for 5 months in a curing room controlled at a temperature of 70 F. and relative humidity of 50 per cent. In the later test series the specimens were tested immediately after 28 days of fog curing. The results of these three series of tests are shown in the accompanying Table III and Fig. 5.

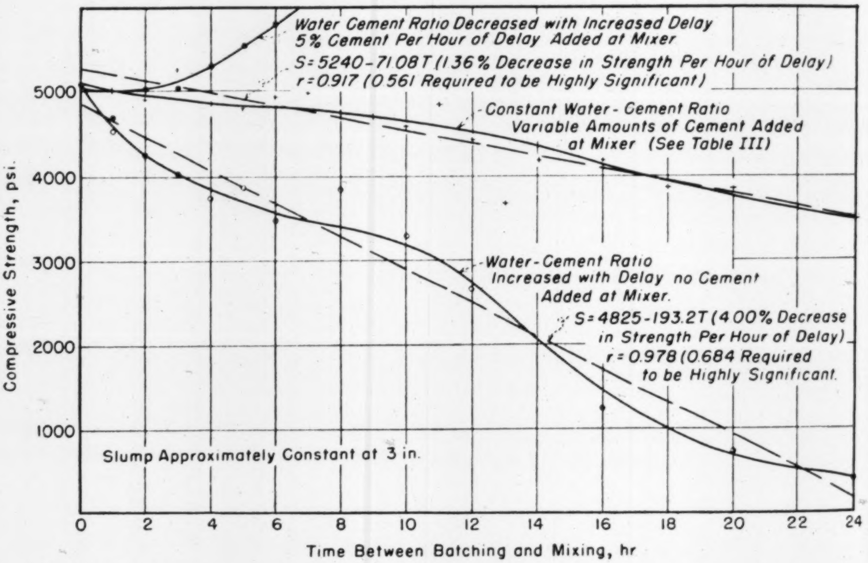


FIG. 5.—Effect of Delay in Mixing of Batched Moist Aggregate and Cement on the Compressive Strength of Concrete Fog-Cured for 28 days.

In series A the cement content was increased by 5 per cent for each hour of delay between batching and mixing. The additional cement was added when the mixer was charged. Mixes were made at 1-hr. intervals for delays up to 6 hr. The 6 by 12-in. cylinders made from this series of mixes showed very little difference in strength for the first 3 hr. of delay. However, there was an appreciable increase in strength for the cylinders made from the batches delayed 4, 5, and 6 hr. The durability of the concrete from each mix in this series was about the same. Although the specimens from the mixes with longer delays showed slightly greater resistance to expansion during the first 50 cycles of freezing and thawing, all of the specimens suffered about 25 per cent weight loss at 100 cycles.

In series B the materials and testing procedures were similar to those described in the original paper for test set No. 3, with the exception that the cement used in series B was slower in setting and the tests were extended for a period of 24 hr. The water requirement for the mixes of series B did not increase as much with delay as was obtained in the original tests. In series B as in the original tests the average strength decrease per hour of delay was about 4 per cent. Immediate mixing of the materials produced concrete with a compressive strength of 5190 psi., whereas a delay between batching and mixing of 12 hr. produced concrete with a compressive strength of 2670 psi. The durability of the concrete of this series did not increase with delay as was obtained in the original tests; however, there was very little decrease in its resistance to cycles of freezing and thawing for the first 6 hr. of delay even though the water-cement ratio was increased with delay.

In series C, a constant water-cement ratio was maintained by adding sufficient additional cement at the time of mixing. The compressive strength tests made with this series of mixes resulted in a decrease in strength of about 1.4 per cent for each hour of delay, even though the water-cement ratio was held constant. It is estimated, for materials comparable to those used in this series, that 60-per cent additional cement would be required after 16 hr. delay and 100-per cent additional cement after 24 hr. to produce concrete of the same strength as obtained by immediate mixing. These results are different from those reported by Mr. Proudley in that they indicate that 50-per cent cement is not sufficient to maintain strength with delays of 16 hr. or over. The difference in the results is due, as Mr. Proudley pointed out, to the methods of tests. In Mr. Proudley's tests only one layer of the cement came in contact with the damp aggregate, whereas, in our tests the cement was thoroughly intermingled with the moist aggregate. Durability tests on specimens from series C are still in progress. Test results after 50 cycles indicate some increase in durability with delays up to 4 hr., and no loss in durability for delays up to 6 hr.

During the discussion from the floor, both Mr. Jackson and Mr. Gonnerman inquired about the increased durability which was indicated for the first 2 hr. of delay in the original tests. We had no satisfactory explanation for this increased durability when the original paper was written, as the unit weights of the concrete do not show a consistent increase in air content with delay. In the later series of tests, air determinations with a pressure-type air meter indicated as much as a 1 per cent gain in air content with delay in mixing, even though the unit weight of the concrete did not decrease accordingly. This discrepancy between unit weight and air meter determinations is believed due to the change in specific gravity of the cement as hydration takes place while the cement is in contact with the moist aggregate. The increase in air content apparently was sufficient to offset the effects of the increase in water-cement ratio on the durability of the concrete. It is noted that the increase in air content with delay is in agreement with the results reported by Mr. Parker in his discussions of this paper.

These tests indicate that the quality of the concrete is maintained for delays up to about 6 hr. by adding cement at the time of mixing in amounts equal to about 3 per cent of the original cement content for each hour of delay. The advisability of keeping batches after 6 hr. delay is questioned because of the increased amount of cement required to maintain the strength. Furthermore, some quick-setting cements might cause trouble after this period of delay. For longer delays, the cement requirement to maintain strength increased very rapidly for each hour of delay, increasing from 60 per cent for 16 hr. to 100 per cent for 24 hr. The bleeding and water requirements of the concrete also increased rapidly for the longer delays, and it is indicated that the drying shrinkage of the concrete would be correspondingly increased.

Mr. Anderegg has brought up a very interesting point regarding the increased shrinkage of mortars which have been allowed to stand before using. Tests made a number of years ago in the laboratories of the Bureau of Reclamation indicated that the resistance to freezing and thawing is increased by long-time mixing. Mortars mixed for 90 min. were found to be about three times more durable than those mixed for 30 min. Although the settlement shrinkage was considerably reduced by the long-time mixing, the drying shrinkage was not appreciably changed. In this case, however, there was some evaporation of water during mixing. Where the mortar does not settle because of (1) reduced time between placing and setting, (2) long-time mixing, or (3) the use of accelerators, it might retain more air which would contribute to its drying shrinkage.

THE EFFECT OF REPEATED LOADING ON THE BOND STRENGTH OF CONCRETE—II*

BY C. W. MUHLENBRUCH[1]

SYNOPSIS

A testing machine to apply repeated loads to bars embedded in concrete, together with certain test results for deformed bars, was described by the author in 1945.[2] The machine has been doubled in speed and modified to permit measurement of slip at both the loaded and unloaded ends of the specimen. The former slips a maximum of 48 per cent more than the latter for hot-rolled bars and 71 per cent more for cold-rolled bars. For undeformed hot-rolled bars subjected to a repeated load equal to 50 per cent of the static pull-out load, the slip ratio (slip at unloaded end divided by slip at loaded end) increased rapidly with increasing cycles of loading. An average maximum slip ratio of about 0.67 was found at one million cycles. Beyond this number of cycles the slip ratio remained constant. The slip ratio limits thus observed varied from 0.52 minimum to 0.78 maximum. Damage-cycle diagrams show the characteristic decrease in static pull-out stresses following various amounts of repeated loading. Similar tests for polished cold-rolled plain bars are reported but varied widely and are not conclusive.

It is concluded that slip of the bar will continue with increasing repetitions of load until failure occurs.

The repeated loading machine for bond specimens, first reported in 1945,[2] has been partially rebuilt to permit measurement of slip at both the loaded and unloaded ends of the specimen during the course of repeated loading. Speed of operation has also been increased. Tests have been confined to plain hot-rolled bars and polished cold-rolled bars.

Object of Investigation:

The purpose of this second stage of the investigation was to determine the influence of repeated loading on the rate of slip at both the loaded and unloaded ends of plain reinforcing bars embedded in concrete.

MATERIALS AND TEST SPECIMENS

Cement and Aggregates Used in Concrete:

Standard Type I portland cement having the physical properties given in Table I was used. The cement was carefully stored in large galvanized cans to prevent segregation and moisture changes.

The Allegheny River sand and gravel used for the fine and coarse aggregates had the gradings given in Table II. The other physical properties given in Table II were determined for the sand and gravel in accordance with the appropriate A.S.T.M. procedures indicated.

As in the previous work the concrete

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Associate Professor of Civil Engineering, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] C. W. Muhlenbruch, "The Effect of Repeated Loading on the Bond Strength of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 824 (1945).

TABLE I.—PROPERTIES OF CEMENT.

Normal consistency, per cent[a]	28.5
Vicat setting time, hr.[a]:	
Initial	3.08
Final	5.25
Tensile strength, 7 days, psi.[a]	325.0
Autoclave expansion, per cent[b]	0.08
Specific surface, sq. cm. per g.[c]	1930.0

[a] Standard Specifications for Portland Cement (C 150 - 46), 1946 Book of A.S.T.M. Standards, Part II, p. 1.

[b] Standard Method of Test for Autoclave Expansion of Portland Cement (C 151 - 43), Ibid., p. 14.

[c] Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (C 115 - 42), Ibid., p. 62.

TABLE II.—GRADINGS AND PROPERTIES OF SAND AND GRAVEL.

Gradings

Allegheny River Sand		Allegheny River Gravel	
Sieve Size	Percentage Passing	Sieve Size	Percentage Passing
No. 4	100	1-½ in.	100
No. 8	98	1 in.	97
No. 16	77	¾ in.	75
No. 30	47	½ in.	37
No. 50	13	⅜ in.	12
No. 100	2	No. 4	2

Physical Properties

	Allegheny River Sand	Allegheny River Gravel
Unit weight, per cu. ft.[a]	101	108
Percentage voids[b]	34	38
Apparent specific gravity[c]	2.50	2.79
Fineness modulus[d]	2.66	7.81

[a] Standard Method of Test for Unit Weight of Aggregate (C 29 - 42), 1946 Book of A.S.T.M. Standards, Part II, p. 474.

[b] Standard Method of Test for Voids in Aggregate for Concrete (C 30 - 37), Ibid., p. 476.

[c] Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 - 42), Ibid., p. 472.
Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 - 42), Ibid., p. 470.

[d] Standard Specifications for Concrete Aggregates (C 33 - 46), Ibid., p. 447.

TABLE III.—PHYSICAL PROPERTIES OF REINFORCING-BAR STEEL.

(Values represent the Average of Three Tests.)

	Hot-rolled	Cold-rolled
Proportional elastic limit, psi.[a]	36 400	52 720
Yield point (determined by drop of beam), psi.[a]	48 200	61 300
Tensile strength, psi.[a]	77 160	72 560
Modulus of elasticity, psi.[b]	30 130 000	29 500 000
Elongation in 8 in., per cent	28.4	19.6
Reduction of area, per cent	51.0	32.4

[a] In accordance with Standard Methods of Tension Testing of Metallic Materials (E 8 - 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 698.

[b] Obtained with an Olsen 2-in. averaging extensometer, having a least reading of 0.0001 in.

mix was proportioned in the ratio of 1:1.5:3.0 by volume and a water-cement ratio of 7.5 gal. per sack was employed. This mix was designed to produce a 28-day compressive strength of 3000 psi., a value selected in order to stay within the capacity of the repeated loading machine. The actual average value for 18 test cylinders was 3040 psi.

Specimens 5 in. on each side were cast in groups of six in the collapsible mold used previously. Curing periods, method of casting, and other variables were likewise kept the same as in the previous work. A total of 10 static load and 11 repeated loading specimens were tested.

Three brass pins, 120 deg. apart, were cast in place in the top surface of the specimens. These pins were drilled out with a No. 60 drill to permit the accurate placing of the pointed legs of the tripod which held the slip measuring dial.

Steel Reinforcing-Bar Material:

The ⅝-in. round reinforcing bars used in these tests conformed to A.S.T.M. Standard Specifications for Billet-Steel Bars for Concrete Reinforcement (A 15 - 39)[3] for the structural grade for the hot-rolled material and to A.S.T.M. Standard Specifications for Cold-Finished Carbon-Steel Bars and Shafting (A 108 - 46)[4] for the 1020 grade for the cold-rolled material. The bars were plain, without any type of surface pattern. The physical properties given in Table III are the average results of extensometer tension tests. All hot-rolled bars were cleaned of foreign matter, but the mill-scale was left in place. Cold-rolled bars were surface polished with a belt sander and thoroughly cleaned before placing in the concrete. Bars with a plain surface were used in an effort to accelerate slip and possible failure due to repeated loading.

---

[3] 1946 Book of A.S.T.M. Standards, Part I-A, p. 91.

[4] 1946 Book of A.S.T.M. Standards, Part I-A, p. 116.

Reinforcing bars were embedded in the concrete within a spiral cage that prevented a splitting failure of the concrete. Each bar was allowed to project 1 in. above the unloaded end of the specimen and 18 in. below. The lower end of the bar was threaded with a $\frac{5}{8}$-in. U. S. Standard thread to permit attachment to the loading device. The length: diameter ratio for this size of bar and the 5-in. embeddment was 8.0.

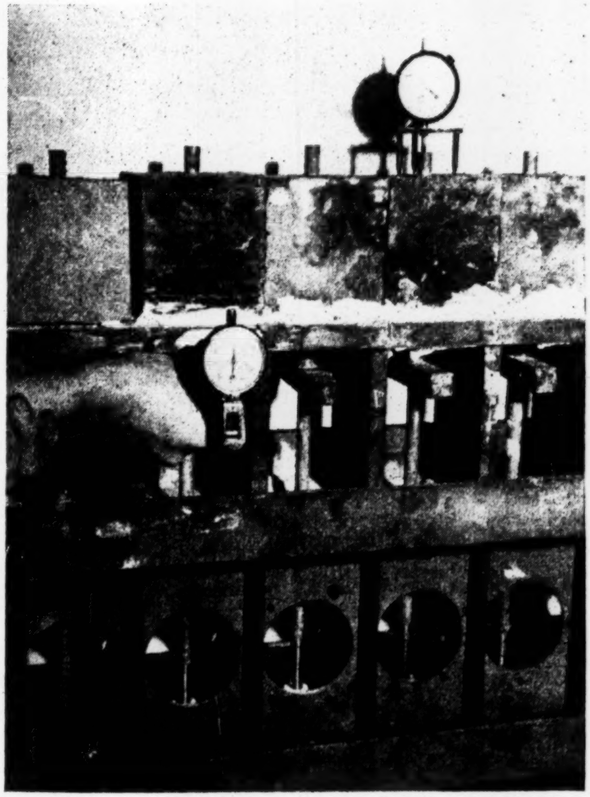


FIG. 1.—Supplementary Supporting Platform for Repeated Loading Specimens. Bar clamps in place and 2-in. Berry strain gage used to determine slip of loaded end of specimen may be seen. Dial indicator on tripod support is shown in place atop a specimen.

TESTING EQUIPMENT

Concrete Test Cylinders:

Compressive strength of the concrete test cylinders was obtained in an Olsen 150,000-lb. capacity universal testing machine. The methods of making and testing the cylinders conformed with A.S.T.M. Tentative Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 44 T)[5] and Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 44).[6]

Slip Measurement:

Static pull-out tests were made in a 50,000-lb. capacity Olsen universal testing machine with the specimen plaster grouted in place on a $\frac{1}{2}$-in. thick steel plate resting on the fixed head. The projecting end of the reinforcing bar was held in V-notch grips to apply load.

---

[5] 1946 Book of A.S.T.M. Standards, Part II, p. 1415.

[6] 1946 Book of A.S.T.M. Standards, Part II, p. 487.

In this series, slip measurements were made in the static pull-out tests in the same manner as in the previous tests except that the brass pins in the unloaded end of the specimen served as reference points for the slip gage. A dial indicator, reading directly to 0.001 in. and by estimation to 0.0001 in., was mounted on a tripod support with pointed legs which fit into the drilled holes in the brass pins.

Slip at the loaded end of the specimen was obtained with a 2-in. Berry strain gage reading to 0.001 in. The two slip gages are shown in place in Fig. 1.

Repeated-Loading Machine:

Two important design changes have been made in the repeated-loading machine. One of these was to double the speed of operation. This was done by driving the speed reducer with a V-belt pulley set having a 1 to 2 speed ratio. The speed of operation was thus increased to 72.92 rpm. or slightly more than 105,000 cycles per day.

The most important change in the original machine was to install a supplementary supporting platform on which the specimens could be placed to permit measurement of slip during repeated loading. This device is shown in detail in Fig. 1. The horizontal 1-in. thick steel plate on which the specimens rest is supported by similar plates bearing on the channel which formerly served as the specimen support. Immediately beneath this supporting plate a 1-in. square bar clamp is fastened to the projecting end of the reinforcing bar. Gage point holes for the Berry strain gage are placed in both ends of this bar clamp and on both sides of the platform. This arrangement permits the slip to be determined as the average of two sides. The Berry strain gage in place is shown in Fig. 1.

Testing procedures were essentially the same as in the former tests. Slip was measured at various cycles of loading for both maximum and minimum repeated loads. The former was maintained constant at 50 per cent of the static pull-out load and the latter at 513 lb. total specimen load[7] or 52 psi. unit bond stress.

TEST RESULTS AND DISCUSSION

Static Pull-Out Tests:

The influence of repeated loading on the static bond stress-slip relationship of the hot-rolled and cold-rolled bar is shown in Fig. 2. A comparison of these data with the previous data for deformed bars shows the plain surface bars to slip considerably more at lower loads than the former. The tendency for the repeated loading to increase the slip at lower loads is apparent from Fig. 2. This figure also shows the erratic behavior of the cold-rolled bars and the comparatively regular behavior of the hot-rolled material.

Damage-Cycle Diagrams:

The damaging effect which prior repeated loading has on the static pull-out strength of both hot and cold-rolled undeformed bars is shown in Fig. 3. The loss at 3 million cycles is 44 per cent. This value is considerably higher than the 31 per cent loss previously reported[2] for the deformed bars at the same number of cycles. The increased damage indicates that the lugs on the deformed bar are helpful in resisting damage due to repeated loading.

---

[7] C. W. Muhlenbruch, "The Effect of Repeated Loading on the Bond Strength of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 837 (1945).

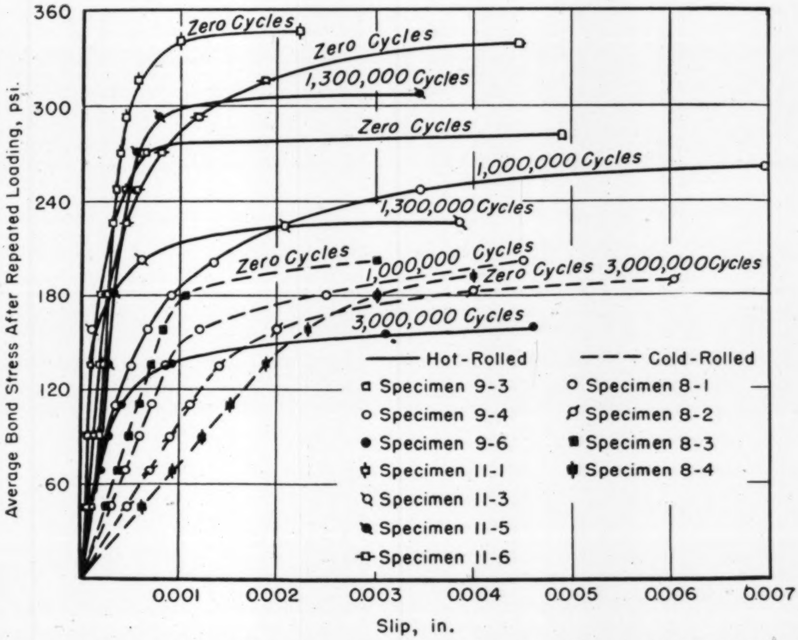


FIG. 2.—Effect of Repeated Loading on the Stress-Slip Relationship For Hot and Cold-Rolled Bars Subjected to Various Cycles of a Repeated Load Equivalent to 50 per cent of the Static Pull-Out Load. Slip is for unloaded end of bar.

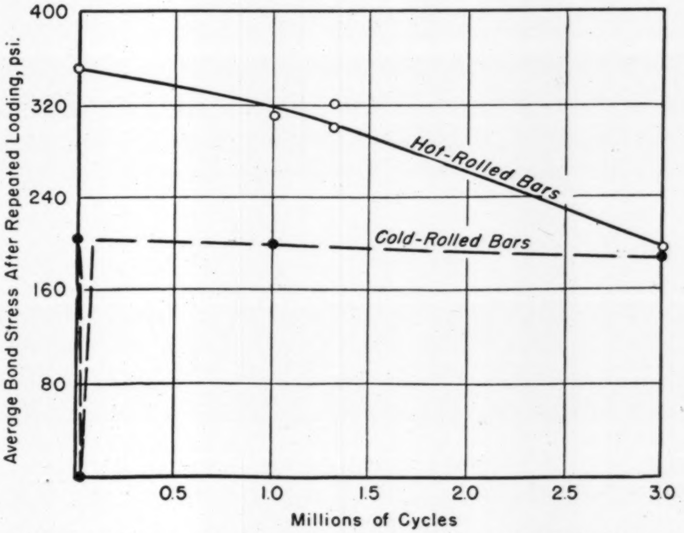


FIG. 3.—Damage-Cycle Diagrams for Specimens Subjected to a Repeated Load Equivalent to 50 per cent of the Static Pull-Out Load.

The erratic behavior of the cold-rolled bars is also evident from Fig. 3. Since several of the specimens pulled out after 200 repetitions of load and others apparently were not damaged by as many as 3 million repetitions it would appear that the damage-cycle data for this material are not conclusive and additional tests must be made.

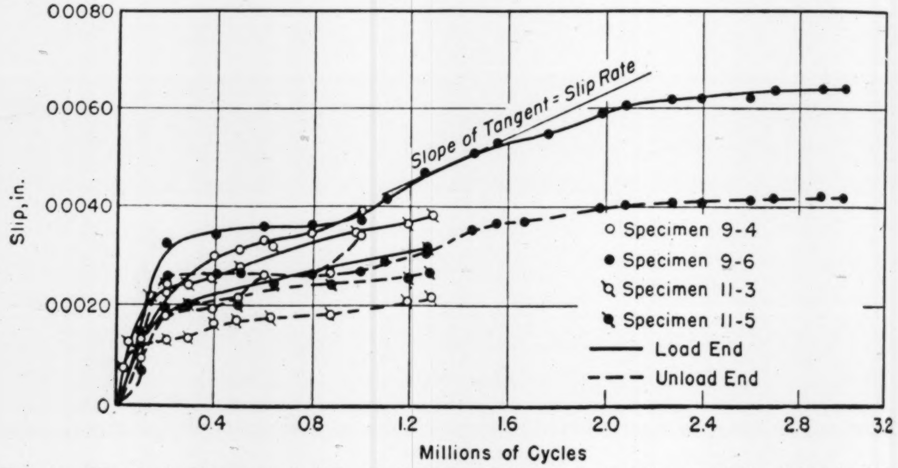


FIG. 4.—Slip-Cycle Diagrams for Hot-Rolled Bars Subjected to a Repeated Load Equivalent to 50 per cent of the Static Pull-Out Load. Slip Rate, in inches per Million Cycles, May be Calculated From the Slope of the Tangent to the Curve.

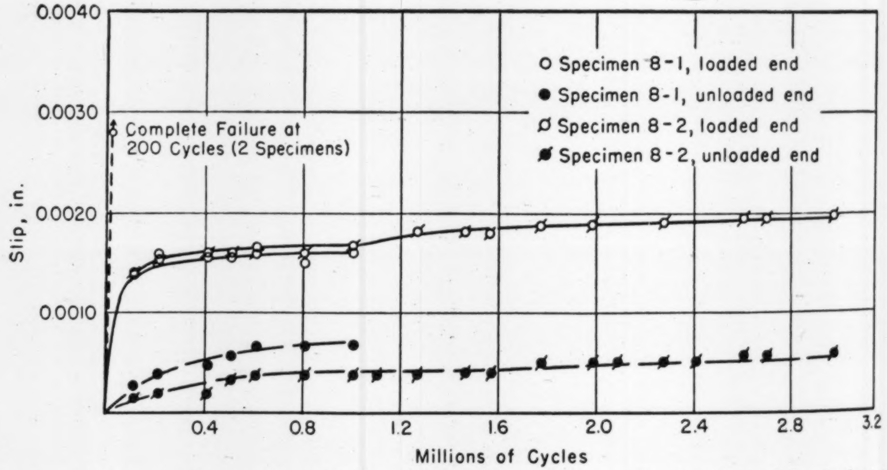


FIG. 5.—Slip-Cycle Diagrams for Cold-Rolled Bars Subjected to a Repeated Load Equivalent to 50 per cent of the Static Pull-Out Load.

Slip Under Repeated Loading:

The most significant data obtained in this series of tests concern the influence of repeated loading on slip. These data are presented in the slip-cycle diagrams of Fig. 4 for the hot-rolled bars and in Fig. 5 for the cold-rolled material, all for a repeated load equal to 50 per cent of the static pull-out load. From these figures it may be seen that both ends of the bar slip rapidly during the first stages of repeated loading. The tangent to the curve at any number of cycles gives the slip rate, in inches slip per million cycles. With increasing repetitions, the slip rate decreased at both the loaded and unloaded ends but had not reached zero at

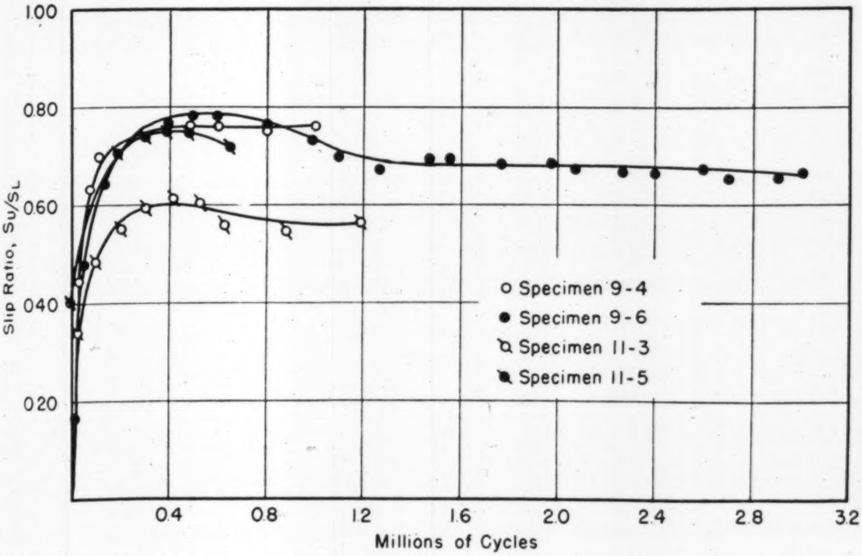


FIG. 6.—The Influence of Cycles of Loading on the Slip Ratio for Hot-Rolled Bars.

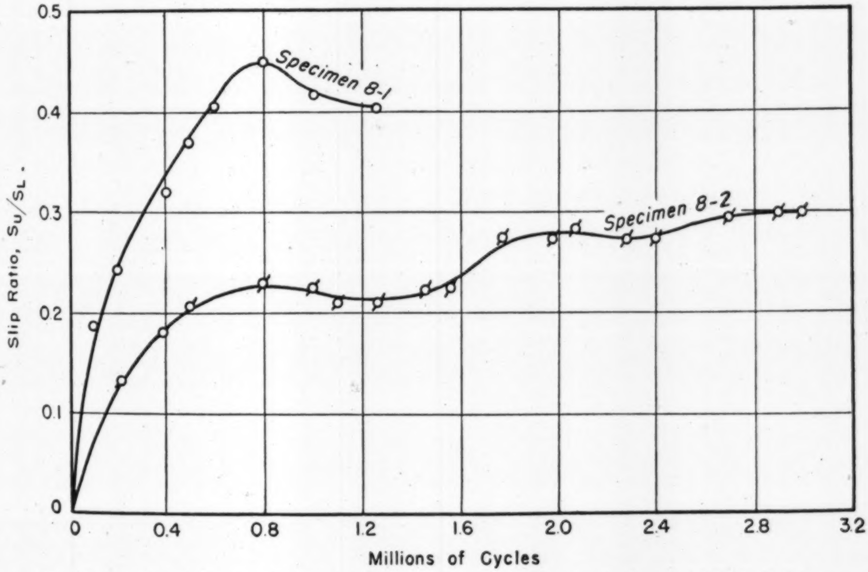


FIG. 7.—The Influence of Cycles of Loading on the Slip Ratio for Cold-Rolled Bars.

3 million cycles. The data also show that the slip rates at either end of the specimen were never equal. From Figs. 4 and 5 the increase in slip at the loaded end of the bar with respect to the unloaded end was found to be a maximum of 48 per cent for the hot-rolled steel and 71 per cent for the polished cold-rolled material. No significance is attached to the fact that those cold-rolled bars which did not fail completely slipped less than the hot-rolled bars. It appears that the polished cold-rolled bars were either gripped properly or not at all, varying so much as to negate any reasonable conclusion regarding their performance.

It was observed from the slip-cycle diagrams that the slip rate of the unloaded end of the bar was never equal to that at the loaded end. This indicates that the bar does not slip uniformly along its length and consequently there is always an uneven distribution of stress along the length of the bar. In an effort to portray the manner in which the breakdown of the bond stress occurs, the term "slip ratio" has been developed. This term is defined as the ratio of the slip at the unloaded end to the slip at the loaded end (S_U/S_L). The slip ratio is compared to repetitions of a load equivalent to 50 per cent of the static pull-out strength in Figs. 6 and 7. From Fig. 6 it may be seen that an average slip ratio of 0.67 was reached after 1,000,000 cycles with a minimum value of 0.52 and a maximum of 0.78. From the curves for the hot-rolled specimens it may be seen that the initial rapid increase in slip at the unloaded end with respect to the loaded end gives evidence of decreasing peak bond stresses at the loaded end as the specimen adjusts itself to the applied load. When this rapid failure zone has been passed the slip ratio remains constant. This stability indicates that the bar is slipping at a rate such that the unloaded end would never slip as much as the loaded end. Theoretically the bar would be slipping uniformly along its length when the slip ratio was 1.0. As long as the slip ratio is less than this value the bar will not slip uniformly. Since the maximum slip ratio observed was 0.78 it follows that slip will continue throughout repeated loading and apparently until the bar pulls out completely. The actual slip rate at the number of cycles representing stability can be obtained from the slip-cycle diagrams of Figs. 4 and 5.

It is seen from Fig. 6 that at about 400,000 cycles the slip ratio is slightly greater than the slip ratio when stability is reached. The reason for the slight decrease in the rate of slip is not apparent.

Figures 5 and 7 show that the polished cold-rolled bars continue to slip with increasing repetitions of load and do not reach a condition of stability. This observation indicates that the slight projections and surface irregularities of the hot-rolled material are quite helpful in maintaining resistance to repeated loading bond failure.

CONCLUSIONS

The data presented as a result of this study indicate that the following conclusions are justifiable for the test conditions and for a repeated load equivalent to 50 per cent of the static pull-out bond strength:

1. Undeformed reinforcing bars slip considerably more and at lower repeated stresses than deformed bars.

2. Three million repetitions of load will reduce the static pull-out strength of a hot-rolled reinforcing bar 44 per cent. A comparison of this damage with the 31 per cent previously reported for deformed bars indicates that the lugs

on the deformed bars aid in resisting damage due to repeated loading.

3. The loaded end of an embedded reinforcing bar slips faster than the unloaded end. With increasing repetitions the slip rate remains greater for the loaded end and may be 48 per cent more than the unloaded end for hot-rolled steel and 71 per cent more for polished cold-rolled steel.

4. The slip ratio for hot-rolled steel increases with increasing repetitions of load, reaching stability at an average value of 0.68. The slip ratio for polished cold-rolled steel continues to increase with increasing cycles of load. The data for this material were erratic, however, and not conclusive.

5. A reinforcing bar will continue to slip with increasing repetitions of load until complete failure by actual pull-out occurs.

Acknowledgment:

The author wishes to acknowledge the work of R. J. Kindl and J. Stein, former seniors in the Department of Civil Engineering of the Carnegie Institute of Technology, who carried out these tests in connection with a senior thesis program under the supervision of the author.

DISCUSSION

Mr. C. P. Siess.[1]—In these tests two measurements have been made: slip at the loaded end and slip at the free end. The author has chosen to consider the ratio of these two quantities which he calls the "slip ratio." I should like to suggest another relation between these measurements which perhaps may throw more light on the behavior of pull-out bond specimens subjected to repeated loading.

It is proposed that the difference between the slips at the loaded and unloaded ends be studied instead of the ratio of these slips. Slip at the loaded end is made up of three parts: the stretch in the embedded length of the bar, the compression of the concrete cube, and the slip at the unloaded end. The difference in slip at the two ends may therefore be interpreted as the stretch of the bar plus the compression of the concrete. This quantity may then be used to obtain some picture of the manner in which the center of bond resistance moves along the bar during a repeated-loading test.

Since the load on the bar and the areas of the bar and the concrete are known, the stress in both the bar and the concrete at the loaded end may be computed. From the moduli of elasticity, the strains corresponding to those stresses may also be computed. Since the difference in slip represents the total deformation of the bar and concrete, it is thus possible to determine, at least approximately, the distance from the loaded end of the bar to the center of bond resistance.

Knowledge of the location of the center of bond resistance and of the manner in which that center shifts during the course of the test should be very helpful in picturing the distribution of the bond stress along the bar and the changes in that distribution resulting from progressive failure under repeated loading.

Chairman F. E. Richart.[2]—As the author noted at the beginning, these tests of plain bars are interesting and furnish information regarding the failure of such bars under repeated bond stress. However, the deformed bar is so universally used now in this country that similar tests on deformed bars would be more useful to engineers. There are a number of new or improved deformed bars now on the market, and if these tests are continued, I hope some of these deformed bars will be included in the program. The effect of repeated loading on bond is still far from being fully known and it is to be hoped that this line of study will be continued.

Mr. C. W. Muhlenbruch (*author's closure*).—An analysis of the over-all behavior of the bar in an effort to isolate the elastic deformations leads to the need for charting the progress of the failure along the bar. The scatter of data presently available for this purpose is so great there does not appear to be any rational explanation of it. In a further attempt to measure the progress of the failure, plans have been developed to use a hollow bar, inserting the electric strain gages in the cavity and measuring the stresses in the bar along its length. This type of bar must of necessity have a lot of bond area. At the present time the machine is being redesigned to accomodate the larger bars.

---

[1] Special Research Assistant Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

It is hoped that future studies will provide data for the path of failure as it moves along the bar so that a complete analysis can be substantiated.

CHAIRMAN RICHART.—Tests are now being made at one laboratory in a manner similar to the one Mr. Muhlenbruch proposes. In this case, a deformed bar is split longitudinally and a space hollowed out in the center in which electric gages are mounted. The two parts of the bar are then securely fastened together and the bar is embedded in a bond specimen.

It might be noted, too, that tubular bars have been used in bond tests, both in this country and in England. However, they have the disadvantage of a non-deformed surface, as well as unusual ratios of bond to tension area.

RESEARCH ON CONCRETE DURABILITY AS AFFECTED BY COARSE AGGREGATE*

By Harold S. Sweet[1]

Synopsis

This paper summarizes the results of laboratory studies undertaken to establish the characteristics of aggregates causing concrete pavement deterioration and of those having satisfactory performance in pavements in Indiana. Extensive field surveys have established previously the relationship in Indiana between concrete pavement performance, in terms of blowups and concrete deterioration, and the coarse aggregate component of the concrete.

The physical properties of 16 coarse aggregate materials are described and the results of laboratory freezing-and-thawing tests on concrete containing these materials are presented. It is concluded, on the basis of the comparison of field performance, aggregate characteristics, and resistance of laboratory concrete to freezing and thawing, that freezing-and-thawing tests on concrete beams containing the aggregate can be used to differentiate materials with good field performance from those with poor performance. The other aggregate properties which were investigated showed no consistent relationship with field performance with the exception of the factor of pore volume and size; the volume of voids smaller in diameter than 0.005 mm., expressed as a ratio to the volume of solids, was less than 0.06 for the aggregates with good field performance and greater than 0.10 for the aggregates with bad service records.

The occurrence of occasional failures of concrete due to causes other than the application of external loads has been a subject of concern for many years. A variety of causal factors has been identified in the many investigations which have been undertaken to establish the reasons for such failures. In concrete pavement surveys made in Indiana, it was found that excessive expansion leading to blowups, and disintegration in the form of mapcracking, were associated with the use of certain coarse aggregates in the concrete (**34**)[2]. It was the purpose of this investigation to determine the reasons for the observed field performance and to develop methods for the identification of good and bad performance materials. To this end, samples of stone and gravel with known service records were obtained from sources throughout the state. Physical properties of each material were determined, including the natural moisture content, microscopic characteristics, void characteristics, and resistance to sodium sulfate and unconfined freezing-and-thawing tests. The aggregates also were incorporated in concrete beams which were subjected to cycles of freezing and thawing. Since a number of field and laboratory studies have demonstrated the importance of degree of saturation

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Research Engineer, Joint Highway Research Project, Purdue University, Lafayette, Ind.—now at University of Wyoming, Laramie, Wyo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1014.

in freezing-and-thawing tests, this factor was given primary consideration with respect to both the aggregate and mortar components of the concrete.

TABLE I.—SUMMARY OF FIELD PERFORMANCE DATA.

Aggregate Source No.	Age of Pavement, (years)	Number of Projects	Total Mileage	Blowups per mile		Degree of Deterioration	Remarks
				Average	Range		
CLASSIFIED AS GOOD PERFORMANCE							
1-1S	15	1	7.9	0	....	None	[a]
67-2S	3	1	4.2	0[b]	....	None	[a]
	11 to 13	3	18.5	0	....	None	[a]
	3	2	7.2	0[b]	....	None	Used with 67-1S
	11 to 14	3	32.3	0	....	None	Used with 67-1S
	3 to 9	3	8.6	0[b]	....	None	With other aggreg.
	10 to 14	2	10.2	0	....	None	With other aggreg.
90-1S	6	2	12.2	0[b]	....	None	[a]
	10 to 11	2	10.9	0	....	None	[a]
	6	1	5.7	0[b]	....	None	Used with 48-2G
	11 to 14	3	23.2	0.4	0 to 1.27	None	With other aggreg.
CLASSIFIED AS BAD PERFORMANCE							
9-1S	10 to 14	10	55.8	5.8	1.9 to 11.7	Severe	[a]
	15 to 18	7	38.9	9.5	1.8 to 25.3	Severe	[a]
	11 to 15	4	22.2	2.3	0.2 to 10.0	Severe	With other aggreg.
35-2S	11	1	3.9	0	....	Severe	[a]
	15 to 16	4	16.2	5.0	2.8 to 11.5	Severe	[a]
	14 to 15	2	17.6	1.0	0.6 to 1.7	Severe	With other aggreg.
47-2S	11 to 12	2	13.3	1.1	0.9 to 1.3	Severe	[a]
	11	1	6.4	0.5[b]	....	Severe	[a]
82-1G	15 to 20	5	32.3	3.2	1.4 to 5.5	Severe	[a]
	2 to 8	3	12.3	0[b]	....	None	With other aggreg.
	11	1	18.4	0.7	....	Moderate	With other aggreg.
	20	4	24.1	1.2	0 to 4.3	Moderate	With other aggreg.
UNCLASSIFIED							
3-1S	3 to 7	3	24.4	0[b]	....	Severe	[a]
	8	1	14.6	0[b]	....	Severe	Used with 40-3S
23-4S	....	0	0	....	....	....	
38-2S	....	0	0	....	....	....	
40-3S	8	1	14.6	0[b]	....	Severe	Used with 3-1S
54-1S	....	....	....	....	....	....	
71-1S	....	....	....	....	....	....	

[a] The only coarse aggregate used in these projects.
[b] Expansion joints were used. Also note the age of the pavenent.

COARSE AGGREGATE PHYSICAL PROPERTIES

The field performance records for the materials tested are summarized in Table I. Three sources with good performance records and four sources with poor performance records are included. In addition, data on materials from six other sources are reported although definite performance records have not been established. Since certain ledges in some of the quarries have been investigated separately, a total of 16 different aggregate materials is represented. The aggregate sources selected for test were chosen so as to represent as many different types of material, geologically and geographically, as possible. For example, numerous quarries are located in the same rock strata as is source 67-2S, and all have excellent performance records. Since the materials are also lithologically similar, only one material from this group, 67-2S, has been investigated. Similar considerations were followed in selecting the other aggregate sources. Brief descriptions of each of the aggregates are given in Table II. More detailed data on field performance, descriptions of the quarries, and mineralogical constituents are given in reference (**25**).

TABLE II.—DESCRIPTIONS OF AGGREGATE MATERIALS.

Aggregate Source	Probable Geological Origin	Fracture	Hardness (Moh's Scale)	Description	Degree of Saturation, Quarry-Wet
			GOOD FIELD PERFORMANCE		
1-1S	Liston Creek—Silurian	Irregular	4.5	Brown-medium-grained dolomitic ls	0.66
67-2S	St. Genevieve—Mississippian	Semi-conchoidal	3	Light grey medium-grained ls	0.33
90-1S	Reef phase of Liston Creek—Silurian	Irregular	4	Light blue-grey medium grained dolomitic ls with many macroscopic voids	0.51
			BAD FIELD PERFORMANCE		
9-1S	Kokomo—Silurian	Irregular	3.5	Buff fine-grained dolomitic ls with very fine laminations	0.94
35-2S Ch	Liston Creek—Silurian	Conchoidal	7	White to light-grey chert	1.00
47-2S	St. Louis—Mississippian	Semi-conch.	3	Brown medium-grained ls	....
82-1G			——	Brown-stained river gravel[a]	1.00
			UNCLASSIFIED PERFORMANCE		
3-1S "D"	Jeffersonville—Devonian	Irregular	3	Light-grey to white fine-grained dolomitic ls	0.97
3-1S "H"	Jeffersonville—Devonian	Irregular	3	White fine-grained dolomitic ls with very fine laminations.	0.96
23-4S	Upper Mississippian	Conchoidal	7	Blue dense chert	....
35-2S Ls	Liston Creek—Silurian	Irregular	3.5	Grey-brown medium-grained dolomitic ls	0.93
38-2S	Reef phase of Liston Creek—Silurian	Irregular	3.5	Light yellow to white fine-grained dolomitic ls with many fine macroscopic voids	low
40-3S	Jeffersonville—Devonian	Irregular	3.5	Light brown medium-grained dolomitic ls with many fine laminations	0.95
54-1S "A"	Upper Mississippian	Irregular	3	Dark-grey fine-grained ls	
54-1S "B"	Upper Mississippian	Irregular	3	White coarse-grained chalky ls	
71-1S	Jeffersonville-Devonian	Irregular	3	Grey to white medium-grained ls	

[a] Lithological composition of ¾ to 1-in. size: 43 per cent chert; 19 per cent quartz and quartzite; 11 per cent granite; 15 per cent other igneous and metamorphic; 12 per cent limestone, sandstone, and shale.

The column in Table II entitled "Degree of Saturation, Quarry Wet" refers to the ratio of the volume of void-space filled with water to the total internal void-volume of the rock. This was determined by obtaining samples of the aggregate immediately after a quarry wall had been dynamited and sealing them in jars which were brought back to the laboratory for determination of the moisture content and apparent and true specific gravity of the rock. In the case of the gravel, the material was sampled immediately after being pumped from the river and was kept under water until tested in the laboratory. The degree of saturation was calculated from the relationship between absorption, apparent specific gravity, and true specific gravity. The degree of saturation in the natural state was considered to be of importance since it is known that aggregate materials are frequently incorporated in concrete within a few hours of their quarrying.

Specific Gravity and Absorption:

A number of determinations of apparent and bulk specific gravity were made for each aggregate. The test procedure used duplicated, essentially, that specified in A.S.T.M. Method C 127 – 42,[3] except that the sample sizes were less than the 5000 g. specified; a torsion balance having a capacity of 4000 g. with a sensitivity of 0.1 g. was employed. The aggregate samples were tested after immersion for 24 hr. and also after evacuation and saturation. The results of these tests are summarized in Table III. Included are the averages and ranges in apparent specific gravity (2, p. 40), bulk specific gravity on a saturated surface-dry basis, and percentage absorption for each of the saturation conditions.

---

[3] Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 - 42), 1946 Book of A.S.T.M. Standards, Part II, p. 470.

TABLE III.—ABSORPTION AND SPECIFIC GRAVITY.

Aggregate Source	By 24-hr. Immersion						By Evacuation and Saturation						No. of Tests
	Specific Gravity				Percentage Absorption		Specific Gravity				Percentage Absorption		
	Apparent		Bulk-Wet				Apparent		Bulk-Wet				
	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range	
GOOD FIELD PERFORMANCE													
1-1S	2.78		2.72		1.4		2.83	2.82-2.84	2.73	2.72-2.73	2.2	2.2- 2.2	3
67-2S	2.72	2.70-2.74	2.68	2.63-2.70	0.7	0.4-1.2	2.72	2.70-2.74	2.68	2.64-2.70	0.8	0.5- 1.3	7
90-1S	2.70	2.66-2.73	2.60	2.56-2.64	2.4	2.2-3.0	2.82	2.81-2.85	2.64	2.55-2.68	3.9	3.0- 6.5	9
BAD FIELD PERFORMANCE													
9-1S	2.68	2.60-2.74	2.44	2.38-2.50	6.0	4.8-8.5	2.86	2.84-2.88	2.50	2.46-2.53	8.3	6.4-10.4	18
35-2S Ch					5.9	4.9-6.5	2.65	2.62-2.67	2.44	2.42-2.46	6.2	5.1- 6.8	8
47-2S	2.69	2.69-2.70	2.62	2.61-2.62	1.7	1.6-1.9	2.72	2.72-2.73	2.62	2.61-2.63	2.4	2.1- 2.9	3
82-1G	2.60	2.58-2.62	2.51	2.49-2.53	2.1	1.8-2.4	2.64	2.62-2.65	2.53	2.50-2.55	2.6	2.4- 2.9	9
UNCLASSIFIED PERFORMANCE													
3-1S "D"	2.71	2.70-2.74	2.48	2.47-2.50	5.7	4.6-7.2	2.84	2.82-2.85	2.52	2.47-2.55	7.4	6.2- 9.1	11
3-1S "H"	2.78	2.77-2.79	2.56	2.54-2.57	4.8	4.6-5.4	2.83	2.78-2.85	2.57	2.55-2.60	5.8	4.5- 6.3	7
23-4S	2.58	2.57-2.58	2.48	2.48-2.48	2.3	2.3-2.3	2.62	2.60-2.62	2.50	2.49-2.50	2.8	2.8- 2.9	4
35-2S Ls					2.9		2.84	2.80-2.85	2.69	2.66-2.71	3.0	2.7- 3.9	4
38-2S	2.66		2.49		4.3		2.84	2.78-2.86	2.55	2.55-2.56	6.3	5.4- 6.8	4
40-3S	2.75		2.62		3.0		2.84	2.82-2.84	2.65	2.64-2.65	4.0	3.9- 4.2	2
54-1S "A"	2.71	2.70-2.72	2.66	2.62-2.68	1.3	0.8-2.0	2.72	2.71-2.72	2.67	2.63-2.69	1.4	0.9- 2.1	6
54-1S "B"	2.62	2.62-2.62	2.47	2.47-2.48	3.9	3.7-4.0	2.72	2.72-2.72	2.50	2.48-2.51	5.3	5.0- 5.8	3
71-1S	2.72		2.61		2.2		2.73	2.71-2.74	2.61	2.61-2.61	2.6	2.3- 2.9	2

A vacuum system was used to obtain more complete saturation of the aggregate in connection with specific gravity and absorption tests and with preparation of the aggregate for incorporation into the concrete. The vacuum system consisted of a sealed chamber connected by individual valves to a manometer, a container of water, a vacuum pump, and an aspirator. Dry aggregate was placed in the vessel and evacuated at pressure of 1.5 to 2.5 cm. of mercury for a period of 1 hr., after which the water was admitted to the vessel with the vacuum still maintained. After the aggregate was covered with water, the vessel was opened to atmospheric pressure. A series of tests was conducted to determine the effect of different pressures and durations of evacuation on the degree of saturation of aggregate 9-1S. For this material, the increase in saturation was negligible when the evacuation pressure was reduced from 2.0 cm. to 0.2 cm. of mercury. In either case a degree of saturation of approximately 97 per cent was obtained for the samples tested. It was concluded that a pressure of 2.0 cm. of mercury for a duration of one hour was adequate for the purposes of this investigation, and this pressure, rather than a lower one, was used because it could be attained readily through the use of an aspirator, and the problem of keeping water vapor out of the vacuum pump could be eliminated.

True Specific Gravity and Volume of Voids:

TABLE IV.—TRUE SPECIFIC GRAVITY AND DEGREE OF SATURATION OF AGGREGATES.

Aggregate Source	True Specific gravity			Ratio: $\frac{\text{Vol. of Voids}}{\text{Vol. of Solids}}$		Degree of Saturation: $\frac{\text{Vol. of Water}}{\text{Vol. of Voids}}$			
						24-hr. Immersion		Evacuation	
	Avg.	Range	Number of Tests	Avg.	Range	Avg.	Range	Avg.	Range
				GOOD FIELD PERFORMANCE					
1-1S	2.87		1	0.075	0.072–0.081	0.55		0.82	0.78–0.86
67-2S	2.77	2.76–2.78	8	0.039	0.029–0.066	0.51	0.30–0.75	0.55	0.35–0.62
90-1S	2.87	2.85–2.89	15	0.131	0.089–0.206	0.52	0.43–0.54	0.86	0.83–0.98
				BAD FIELD PERFORMANCE					
9-1S	2.87	2.87–2.88	4	0.242	0.191–0.312	0.71	0.66–0.78	0.98	0.96–0.98
35-2S Ch	2.66		2	0.169	0.136–0.181	0.93		0.98	0.97–0.98
47-2S	2.74		1	0.067	0.063–0.100	0.71	0.07–0.78	0.90	0.89–0.90
82-1G	2.67	2.67–2.68	2	0.082	0.075–0.087	0.67	0.58–0.81	0.86	0.74–0.95
				UNCLASSIFIED PERFORMANCE					
3-1S "D"	2.86		2	0.218	0.183–0.265	0.75	0.69–0.83	0.97	0.96–0.98
3-1S "H"	2.88	2.87–2.90	3	0.179	0.154–0.201	0.80	0.74–0.83	0.90	0.79–0.96
23-4S	2.65		1	0.087	0.086–0.088	0.69		0.87	
35-2S Ls	2.87	2.86–2.88	2	0.097	0.084–0.133			0.89	0.76–0.92
38-2S	2.89	2.88–2.90	3	0.205	0.200–0.215	0.59	0.58–0.60	0.91	0.78–0.96
40-3S	2.87	2.86–2.88	2	0.128	0.123–0.133	0.66	0.65–0.68	0.91	0.86–0.94
54-1S "A"	2.74	2.71–2.76	6	0.046	0.022–0.108	0.76	0.51–1.00	0.85	0.80–1.00
54-1S "B"	2.73	2.72–2.74	3	0.148	0.138–0.166	0.72	0.69–0.73	0.97	0.95–1.00
71-1S	2.78	2.76–2.79	5	0.089	0.075–0.099	0.74	0.70–0.81	0.85	0.82–0.92

Samples of material were powdered in a mortar and pestle to pass a No. 50 sieve. The specific gravity of this powdered material was determined using the procedure for determination of specific gravity of soils (**7**). The values obtained are shown in Table IV. Also shown in this table, for each aggregate, is the volume of voids as a ratio of void volume to volume of solids. This ratio was calculated from the following relationship between apparent specific gravity, absorption, and true specific gravity (**25**):

$$\frac{\text{Volume of Air}}{\text{Volume of Solids}} = \frac{\text{True Specific Gravity} - \text{Apparent Specific Gravity}}{\text{Apparent Specific Gravity}}$$

$$\frac{\text{Volume of Absorbed Water}}{\text{Volume of Solids}} = \frac{\text{Percentage Absorption} \times \text{True Specific Gravity}}{100}$$

$$\frac{\text{Total Void Volume}}{\text{Volume of Solids}} = \frac{\text{Volume of Air} + \text{Volume of Absorbed Water}}{\text{Volume of Solids}}$$

The values of this ratio may be considered to be the actual void volume associated with one cubic unit of solid volume and will be termed simply the volume of voids.

Microscopic Analysis of Voids:

Detailed data on the size and distribution of pores in the aggregate were obtained by means of thin sections and polished sections of the aggregates. The thin sections were examined by transmitted polarized light, and drawings of the voids were made using a camera-lucida attachment. The polished sections were first stained by rubbing the polished surface with powdered red copper oxide (cuprous) and wiping off the excess pigment. The stained surfaces were examined in reflected light and drawings of the void structure were made with the camera-lucida attachment. This technique has been described with reference to determining the air content of hardened concrete by Verbeck (**28**). The highest magnification used was 100; the smallest voids so determined were approximately 0.005 mm. in diameter. The area of the voids was determined by planimetering the drawings. The total void volume of the polished specimens was determined from the specific gravity and absorption of the individual specimens. They were immersed in water for 24 hr., weighed in water suspended from the arm of an analytical balance, and then surface-dried and weighed in air on the analytical balance. After oven-drying and weighing, the apparent specific gravity and absorption of the individual pieces were calculated. Total void volume was calculated from the relationships previously described using the average true specific gravity value shown in Table IV.

TABLE V.—MICROSCOPIC DETERMINATION OF AGGREGATE VOID CHARACTERISTICS.

Aggregate	Volume of Voids[a] Larger Than 0.005 mm.	Volume of[a] Total Voids	Volume of Voids[a] Smaller Than 0.005 mm.
GOOD FIELD PERFORMANCE			
1-1S	0.02	0.07	0.05
67-2S	0.02	0.04	0.02
90-1S	0.09	0.10	0.01
BAD FIELD PERFORMANCE			
9-1S	0.11	0.23	0.12
35-2S Ch	0.06	0.17	0.11
47-2S	0	0.11	0.11
UNCLASSIFIED PERFORMANCE			
3-1S "D"	0.02	0.16	0.14
3-1S "H"	0	0.20	0.20
23-4S	0.02	0.07	0.05
35-2S Ls	0.09	0.11	0.02
38-2S	0.18	0.20	0.02
40-3S	0	0.12	0.12
54-1S "B"	0.06	0.14	0.08
71-1S	0.02	0.09	0.07

[a] Expressed as a ratio to the volume of solids: $\frac{V_v}{V_s}$.

TABLE VI.—RESULTS OF SODIUM SULFATE AND UNCONFINED FREEZING-AND-THAWING TESTS ON COARSE AGGREGATES

Aggregate Source	Percentage Loss in Sodium Sulfate Test		Percentage Loss in 50 Cycles Freezing and Thawing	
	5 cycles	10 cycles	24-hr. Immersion	Evacuation
GOOD FIELD PERFORMANCE				
1-S	3.0		3.3	4.8
67-2S	2.5		3.0	4.7
90-1S	2.9		5.7	4.2
BAD FIELD PERFORMANCE				
9-1S	10.8		6.8	8.5
35-2S Ch	17.2		32.4	63.7
47-2S	6.6	12.8	7.2	20.7
82-1G	0.7	2.3	10.0	27.2
UNCLASSIFIED PERFORMANCE				
3-1S "D"	12.7		5.7	14.4
3-1S "H"	10.4	42.6	5.1	10.8
23-4S	3.7		6.3	13.3
38-2S	5.7		7.2	4.3
40-3S	3.4		2.6	14.0
54-1S "A"	16.9	38.9		15.9
54-1S "B"	21.3	58.6	8.4	7.8
71-1S	17.8	48.1	11.8	6.1

Table V shows the volume of voids greater than 0.005 mm. in diameter and the total void volume expressed as a ratio to the volume of solids. The difference between the total void volume and the volume of voids larger than 0.005 mm. is recorded.

Sodium Sulfate and Unconfined Freezing-and-Thawing Tests:

The procedure for the sodium sulfate test corresponded to that outlined in A.S.T.M. Method C 88 – 46 T.[4] The

---

[4] Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 46 T), 1946 Book of A.S.T.M. Standards, Part II, p. 1400.

TABLE VII.—ANALYSES OF CEMENT.

Chemical Analysis, per cent:	
SiO_2	21.02
Al_2O_3	6.55
Fe_2O_3	2.67
CaO	64.80
MgO	1.15
SO_3	1.79
Loss on ignition	1.54
Total	99.52
Compound Composition, per cent:	
3 CaO SiO_2	47.78
2 CaO SiO_2	24.29
3 CaO Al_2O_3	12.83
4 CaO Al_2O_3 Fe_2O_3	8.12
$CaSO_4$	3.04
Free MgO	1.15
Free CaO	0.77
Insoluble Residue	0.20
Na_2O	0.17
K_2O	0.88

Physical Tests:	
Fineness No. 200	97.0
Fineness No. 325	94.7
Surface Area	1865 sq. cm. per g.
Set Initial	3:20
Final	5:15
Autoclave Expansion	0.135%
Normal Consistency	24.0
24 hr. Temp., Fahr.	71
Relative Humidity	95
Tensile Strength, psi.:	
1:3 Sand Briquets:	
1 Day	173
3 Days	373
7 Days	448
28 Days	502
Compressive Strength 2-in. Cubes, psi:	
1 Day	792
3 Days	2247
7 Days	3750
28 Days	5617

percentage losses after five and ten cycles of the treatment are shown in Table VI.

The aggregates were tested in freezing and thawing in both the 24 hr. immersed and evacuated conditions. Approximately 1500 g. of material passing a 1-in. sieve was carefully separated on a $\frac{3}{4}$-in. sieve, oven-dried, and weighed. The samples were immersed for 24 hr. and then placed in a freezer at −8 F. while partially covered with water. After at least three hours in the freezer they were placed in a thawing tank for one to three hours and then the cycle was repeated. After 50 cycles they were oven-dried and again sieved on the $\frac{3}{4}$-in. sieve. The difference between the final weight of material retained and the initial weight, calculated as a percentage of the original weight, is recorded in Table VI as the percentage loss. This same procedure was used for duplicate samples except that the aggregate was evacuated and saturated as described previously prior to the first cycle of freezing and thawing.

CONCRETE TESTS

To establish a cause for the field performances observed, an attempt was made to duplicate, under controlled laboratory conditions, the deterioration of concrete. The laboratory concrete made with aggregates having unsatisfactory performance records was expected to show cracking and growth when subjected to simulated weathering tests. A concrete mix was chosen to approach common highway concrete mixtures as closely as possible:

1. Six bags of cement per cubic yard of concrete. The chemical analysis and physical test data on the cement are shown in Table VII.

2. The absolute volume of fine aggregate was the same for each mix—0.268 cu. ft. of fine aggregate per cubic foot of concrete. The fine aggregate was a standard concrete sand of glacial origin. Its physical properties are shown in Table VIII.

TABLE VIII.—CHARACTERISTICS OF FINE AGGREGATE.

Gradation:

Sieve Size:	Total Percentage Passing
4	99.8
8	89.1
16	66.8
30	35.4
50	12.0
100	2.1

Fineness modulus: 2.95
Unit weight in above gradation: 110.0 lb. per cu. ft.
Apparent specific gravity: 2.72
Bulk specific gravity (dry): 2.60
Bulk specific gravity (wet): 2.67
Absorption, percentage: 1.5
Mortar Tensile Strength: (A.A.S.H.O. Method T-35-35)
Percentage of Standard Ottowa at 7 days: 124
at 28 days: 129

TABLE IX.—MIX DATA FOR CONCRETE WITH EVACUATED AGGREGATE.

Beam Numbers	Cement Factor, bags per cu. yd.	Water-Cement Ratio, by weight	Initial Aggregate Saturation, per cent	Slump, in.	Calculated Percentage of Air, Fresh	Unit Weight, lb. per cu. ft.			28-Day Dynamic Modulus, psi.	28-Day Modulus of Rupture, psi.	28-Day Mortar Saturation, per cent
						Fresh	At Number of Days shown( )				
GOOD FIELD PERFORMANCE											
Aggregate 1-1S:											
1360–1363	6.00	0.51	82	2	2.6	150.2	153.3(4)	153.6(28)	6 260 000	850	73
1364–1365	5.84	0.51	82	2	2.8	150.0	153.2(3)	153.9(28)	6 340 000	...	73
Aggregate 67-2S:											
1114–1119	5.56	0.57		2	2.3	(149.0)		152.4(72)	5 800 000	800	79
1156–1163	5.98	0.55		2	1.3	149.1	150.2(3)	151.4(28)	5 610 000	650	
1638–1640	6.10	0.48		2	1.8	151.5	152.1(11)	153.3(28)	6 050 000	750	61
Aggregate 90-1S:											
1396–1404	6.04	0.47	86	2	1.9	149.8	152.2(2)	152.4(28)	6 090 000	860	51
1411–1412	5.96	0.52	8	3	1.8	149.5	152.1(7)	152.3(28)	5 980 000	...	65
BAD FIELD PERFORMANCE											
Aggregate 9-1S:											
1120–1125	5.57	0.55		2	2.0	145.0		146.4(68)	5 400 000	780	70
1148–1155	6.00	0.52		1	1.5	145.0	145.3(3)	146.0(28)	4 900 000	640	70
1231–1241	5.76	0.50		6	3.6	142.5	146.4(2)	147.4(28)	5 020 000	650	72
1257–1259	5.98	0.49	98	2	1.7	(145.5)	147.0(3)	147.7(28)	5 430 000	...	71
1721–1732	6.03	0.49	95	2	2.2	145.5	147.2(2)	148.3(28)	5 200 000	710	69
Aggregate 35-2S Chert:											
1193–1197	6.03	0.52	98	5		142.8	145.9(2)	147.1(28)	5 050 000	610	
Aggregate 47-2S:											
1278–1280	6.00	0.55	95	3	1.8	(147.0)	147.9(3)	148.9(29)	5 300 000		76
Aggregate 82-1G:											
1164–1171	5.96	0.56		8		145.5	146.4(3)	147.5(28)	5 300 000	460	
1211–1221	6.00	0.52		6		147.0	146.9(3)	147.9(28)	5 740 000	560	
1531–1542											
UNCLASSIFIED PERFORMANCE											
Aggregate 3-1S "D":											
1208–1210	5.76	0.63		7	2.2	142.0	142.7(4)	143.9(28)	4 280 000		76
1327–1331	5.94	0.50	96	2		143.0	145.5(2)	147.4(28)	5 020 000		
1336–1339	6.04	0.47	95	2			147.1(2)	148.3(28)	5 220 000		
Aggregate 3-1S "H":											
1222–1230	6.00	0.49		2	2.0	147.3	147.8(2)	148.8(28)	5 260 000	630	57
1321–1326	6.03	0.49	81	2	3.6	146.0	146.1(2)	148.3(28)	5 020 000	...	70
1332–1335	6.01	0.49	92	2	2.7	145.5	147.6(2)	149.2(28)	5 150 000	...	77
Aggregate 23-4S:											
1512–1519	6.06	0.51	87	2	3.9	143.0		148.1(28)	6 140 000	805	65
Aggregate 35-2SLs:											
1275–1277	5.95	0.50	77	3	1.6	(148.0)	149.0(3)	150.8(32)	5 490 000		80
Aggregate 38-2S:											
1356–1359	6.13	0.46	80	2	2.3	146.3	149.2(4)	149.5(28)	5 480 000	750	58
1366–1367	5.96	0.48	79	5	3.7	144.2	148.4(3)	148.8(28)	5 170 000	...	62
Aggregate 40-3S:											
1198–1207	5.76	0.49		2	4.4	146.0	148.9(3)	149.6(28)	60 000	580	45
1407–1408	5.95	0.57	92	5	2.1	147.0	149.4(7)	150.1(28)	5 420 000	...	82
Aggregate 54-1S "A":											
1457–1461	5.93	0.43	83	2	3.6	149.0		151.9(28)	5 740 000		30
Aggregate 54-1S "B":											
1368–1375	6.01	0.52	97	2	1.8	144.9	146.7(2)	147.5(28)	3 930 000	610	69
1394–1395	6.05	0.52	98	3	2.0	144.5	146.5(4)	146.5(29)	3 780 000		55
Aggregate 71-1S:											
1502–1505	6.10	0.53	85	4		147.5		150.7(30)	5 640 000	800	

3. The absolute volume of coarse aggregate was 0.441 cu. ft. per cubic foot of concrete and was the same for each mix.

TABLE X.—MIX DATA FOR CONCRETE WITH IMMERSED AGGREGATE.

Beam Numbers	Cement Factor, bags per cu. yd.	Water-Cement Ratio, by weight	Initial Aggregate Saturation, per cent	Slump, in.	Calculated Percentage Air, Fresh	Unit Weight, lb. per cu. ft.			28-Day Dynamic Modulus, psi.	28-Day Modulus of Rupture, psi.	Calculated Percentage Mortar Saturation, 28-days
						Fresh	At Number of Days shown ( )				
GOOD FIELD PERFORMANCE											
Aggregate 1-1S:											
1439–1444	6.03	0.47	55	1	3.5	149.0		153.7(28)	6 630 000		62
Aggregate 67-2S:											
1299–1309			(51)	2			153.1(8)	152.8(29)	6 100 000		
Aggregate 90-1S:											
1462–1468	6.10	0.49	45	1	1.2	149.5		151.4(30)	6 000 000		73
BAD FIELD PERFORMANCE											
Aggregate 9-1S:											
1108–1113	5.56	0.57	(71)	3		(143.0)		145.5(75)	5 300 000	650	
1182–1192	5.82	0.56	(71)	6		(143.0)	147.0(2)	148.0(28)	5 220 000	640	
1310–1319			(71)	2			147.6(8)	147.9(29)	5 070 000		
Aggregate 47-2S:											
1248–1250	6.05	0.55	(71)	1	1.7	(147.0)	147.9(2)	149.0(28)	5 360 000		73
Aggregate 82-1G:											
1242–1244	6.06		77	6	3.9	(146.0)	147.0(3)	148.0(28)	5 520 000		
1483–1494	6.22	0.48	64	7	1.9	147.0		147.8(30)	5 840 000	750	60
1520–1530	6.26	0.46	87	3	1.6	146.5		147.6(28)	5 760 000	700	52
UNCLASSIFIED PERFORMANCE											
Aggregate 3-1S "D":											
1251–1253	6.15	0.42	83	3	3.1	(144.0)	145.7(2)	146.6(29)	5 090 000		50
Aggregate 3-1S "H":											
1254–1256	5.96	0.54	81	5	1.2	(145.5)	147.1(2)	147.6(29)	5 020 000		
Aggregate 38-2S:											
1445–1451	6.12	0.51	59	1	1.5	145.0		149.7(29)	5 920 000		
Aggregate 40-3S:											
1245–1247	5.70		78	1		(146.0)	149.0(3)	150.3(28)	5 780 000		
Aggregate 54-1S "B":											
1469–1475	5.50	0.52	(72)	2	2.7	142.5		146.1(30)	3 680 000		74
Aggregate 71-1S:											
1476–1482	6.00	0.50	73	2	2.7	146.5		150.5(30)	5 490 000		74

4. Sufficient mixing water was used to obtain a slump of 2 to 4 in.

A total of 33 batches of concrete with the 16 aggregates in an evacuated and saturated condition was made, and 16 mixes were made with the aggregates in an immersed condition. In addition, 30 batches of concrete were made using different combinations of aggregates with good and bad field performance records. From this concrete a total of 369 beams was made, 3 by 4 by 16 in. and 3 by 4 by 24 in. in size.

Preparation of Concrete:

The coarse aggregates were separated into different sizes and combined to the following proportions:

¾ in. to 1 in.	25 per cent
½ in. to ¾ in.	25 per cent
⅜ in. to ½ in.	25 per cent
No. 4 to ⅜ in.	25 per cent

The coarse aggregate was weighed room-dry and either immersed or evacuated and immersed for 24 hr. prior to mixing. The water was drained off and the wet aggregate reweighed. From previous absorption determinations, the weight of absorbed water was calculated and subtracted from the water content, the difference being considered mixing water.

The concrete was hand-mixed and slump and unit weight tests were performed. Molding of the beams conformed to the procedures recommended by Withey (**33**).

TABLE XI.—MIX DATA FOR CONCRETE WITH COMBINATIONS OF AGGREGATES.

Beam Numbers	Percentage by weight of Poor Performance Aggregate					Cement Factor, bags per cu. yd.	Water-Cement Ratio	Slump, in.	Calculated Percentage Air, Fresh	Unit Weight, lb. per cu. ft.		28-Day Dynamic Modulus, psi.	Calculated Percentage Mortar Saturation, 28 days
	No. 4–⅜ in.	⅜–½ in.	½–¾ in.	¾–1 in.	Total Coarse Aggregate					Fresh	At 28 days		
COMBINATIONS OF 35-2S CHERT EVACUATED (BAD PERFORMANCE) WITH 67-2S LIMESTONE IMMERSED (GOOD PERFORMANCE)													
1293–1295	0	0	0	10	2.5	5.96	0.56	3	1.9	(148.0)	151.2	5 990 000	76
1296–1298	0	0	10	0	2.5	5.97	0.52	6	2.7	(148.0)	149.8	5 960 000	51
1344–1345	0	0	0	20	5.0	6.13	0.48	6	3.6	147.5	151.1	5 700 000	42
1346–1347	0	0	20	0	5.0	6.21	0.45	2	3.3	148.5	152.2	6 060 000	26
1281–1283	5	5	5	5	5.0	6.00	0.52	4	1.5	(148.0)	150.9	6 100 000	84
1342–1343	5	5	5	5	5.0	6.16	0.47	2	3.5	147.5	152.4	6 050 000	56
1508–1509	5	5	5	5	5.0	6.27	0.45	6	3.3	148.5	151.2	5 860 000	27
1284–1286	10	10	10	10	10.0	6.02	0.52	3	2.2	(148.0)	149.7	6 040 000	60
1340–1341	10	10	10	10	10.0	6.16	0.47	1	3.3	147.5	151.9	6 180 000	46
1287–1289	15	15	15	15	15.0	6.06	0.48	3	2.4	(149.0)	151.1	6 040 000	45
1348–1349	0	0	0	100	25.0	6.17	0.46	2	3.8	146.0	150.0	5 680 000	35
1350–1351	0	0	100	0	25.0	6.11	0.48	4	2.8	147.0	150.8	5 840 000	47
1506–1507	0	0	100	0	25.0	6.13	0.48	2	2.6	147.5	151.1	6 150 000	53
1352–1353	0	100	0	0	25.0	6.07	0.47	2	3.7	146.0	150.2	5 720 000	39
1354–1355	100	0	0	0	25.0	6.07	0.47	2	3.5	146.0	149.7	5 630 000	37
1290–1292	25	25	25	25	25.0	6.06	0.52	3	1.8	(149.0)	150.6	5 920 000	57
COMBINATIONS OF 82-1G (EVACUATED) WITH 67-2S (IMMERSED)													
1378–1379	100	100	0	0	50.0	6.05	0.52	6	1.8	(147.0)	149.9	5 580 000	72
1386–1387	100	100	0	0	50.0	6.08	0.47	6	3.2	(147.0)	150.8	5 920 000	46
1382–1383	50	50	50	50	50.0	6.21	0.44	2	3.3	(147.0)	150.8	5 980 000	28
1392–1393	50	50	50	50	50.0	6.02	0.52	5	2.5	(147.0)	149.7	5 670 000	61
1376–1377	100	100	100	0	75.0	6.06	0.54	6	1.1	(147.0)	149.8	5 870 000	83
1384–1385	100	100	100	0	75.0	6.14	0.46	2	2.5	(147.0)	149.5	5 960 000	39
1380–1381	75	75	75	75	75.0	6.10	0.50	2	1.4	(147.0)	150.4	5 990 000	81
1390–1391	75	75	75	75	75.0	6.05	0.52	2	1.4	(147.0)	149.5	5 770 000	74
COMBINATIONS OF 9-1S (EVACUATED) WITH 67-2S (IMMERSED)													
1266–1268	0	0	0	100	25.0	5.97	0.55	3			150.7	5 840 000	
1269–1271	0	0	100	0	25.0	6.02	0.49	6	2.7	(148.0)	149.9	5 720 000	54
1272–1274	0	100	0	0	25.0	6.06	0.49	3	2.0	(149.0)	151.1	5 750 000	59
1263–1265	25	25	25	25	25.0	5.84	0.60	2			151.0	6 000 000	
1260–1262	50	50	50	50	50.0	5.94	0.53	2	1.4	(147.0)	148.9	5 510 000	76
COMBINATIONS OF 35-2S CHERT (EVACUATED) WITH 35-2S LIMESTONE (IMMERSED)													
1172–1181					15.0	6.0	0.56	6	1.9	146.8	148.5	5 000 000	77

Tests on Hardened Concrete:

After curing in the molds for two days, the beams were removed and the weights in air and immersed in water were determined, which weights redetermined periodically during the curing period and during the freezing-and-thawing tests. At the same intervals, the length and natural frequency of each beam were determined. Equipment and test procedure for determining the natural frequency conformed to that specified in A.S.T.M. Tentative Method C 215 – 47 T.[5]

After 28 days of curing, two or more beams from each batch were subjected to alternate freezing at −8 F. for 16 hr. followed by thawing in water at 60 F. for 8 hr. In general, the beams containing aggregate which had been vacuum-saturated were frozen in air and the beams containing aggregate which had

TABLE XII.—SUMMARY OF FREEZING-AND-THAWING TEST DATA FOR CONCRETE BEAMS WITH EVACUATED AGGREGATE.

Coarse Aggregate	Beam Numbers	Number of Beams	Initial Tests				Start of Freeze			Type of Freeze	Number of Freezing-and-Thawing Cycles			Avg. Percentage Loss[a] in Dynamic Modulus per Cycle of Freezing and Thawing
			Age, days	Percentage Difference[a]			Age, days	Unit Weight, lb. per cu. ft.	Dynamic Modulus, psi.		10 per cent Loss in Dynamic Modulus	30 per cent Loss in Dynamic Modulus	End of Test	
				in Weight	Unit Weight	Dynamic Modulus								
GOOD FIELD PERFORMANCE														
1-1S	1360-61	2	4	−0.10	−0.20	−14	28	153.6	6 250 000	Air	32	76	82	0.41
	1364-65	2	3	−0.15	−0.45	−23	28	153.9	6 340 000	Air	18	69	74	0.58
67-2S	1117-19	3	53	0	0	0	53	152.1	5 730 000	Air	250	295+	295	0.06
	1157-59	3	3	−0.70	−0.92	−30	28	151.6	5 650 000	Air	211	329	343	0.12
	1163	1	3	−0.46	−0.07	−16	7	151.0	4 740 000	Air	100	116	168	0.56
	1638	1	1	0	−0.98	−19	28	153.3	6 050 000	Water	30	75+		0.21
	1639	1	1	−0.14	−0.59	−19	28	152.7	5 940 000	Air	28	75+		0.36
90-1S	1400-02	3	2	−0.43	−0.08	−29	28	152.6	6 230 000	Water	5	7	18	4.78
	1403-04	2	2	−0.20	−0.20	−32	28	151.7	6 000 000	Air	3	7	12	4.38
	1411-12	2	7	−0.08	−0.10	−11	28	152.3	5 970 000	Air	3	12	33	2.60
BAD FIELD PERFORMANCE														
9-1S	1123-25	3	4	−0.54	...	...	58	146.4	5 400 000	Air	3	8	16	4.07
	1149-54	2	3	−1.07	−0.55	−25	28	145.7	4 820 000	Air	11	...	11	1.20
	1153	1	3	−0.65	−0.34	−15	7	145.8	4 170 000	Air	16	26	70	1.31
	1151-2	2	3	−1.00	−0.21	−25	28	145.5	4 900 000	Air	14+	...	14	0.61
	1231-33	3	2	−0.85	−0.75	−36	29	147.4	5 080 000	Air	21	36	47	0.92
	1257-59	3	3	−0.35	−0.44	−28	28	147.7	5 430 000	Air	3	6	14	4.72
	1721-22	2	2	−1.05	−0.68	−33	28	147.8	5 180 000	Air	3	12	12	5.30
	1723-24	2	2	−1.10	−1.15	−33	28	147.8	5 210 000	Water	2	7	8	5.08
35-2S Ch	1193-95	3	2	−0.78	−0.79	−22	28	147.3	5 030 000	Air	1	2	20	12.30
47-2S	1278-79	2	3	−0.73	−0.51	−33	29	148.9	5 300 000	Air	12	36	45	0.91
82-1G	1167-68	2	2	−0.48	−0.68	−35	28	147.7	5 380 000	Air	2	4	12	8.75
	1213-16	4	3	−0.50	−0.74	−31	283	148.4	6 020 000	Air	1	2	9	13.10
	1217-19	3	3	−0.61	−0.74	−25	28	148.2	5 700 000	Air	3	6	24	5.28
UNCLASSIFIED PERFORMANCE														
3-1S "D"	1208-09	2	4	−0.85	−0.904	−26	28	143.8	4 260 000	Air	6	10	24	3.86
	1327-28	2	2	−1.70	−1.22	−27	28	147.5	4 940 000	Air	9	30	46	1.32
	1329-31	3	2	−2.06	−0.47	−28	39	147.2	5 060 000	Air	5	28	37	1.41
	1336-39	4	2	−0.92	−0.77	−27	28	148.3	5 220 000	Air	2	6	13	4.51
3-1S "H"	1229-30	2	2	−0.72	−0.67	−26	28	149.0	5 310 000	Air	4	10	37	2.56
	1321-24	4	2	−1.71	−1.48	−26	39	148.4	5 050 000	Air	6	26	50	1.44
	1325-26	2	2	−1.71	−1.52	−26	28	148.2	5 210 000	Air	8	23	32	1.70
	1332-36	4	2	−1.05	−1.13	−27	28	149.2	5 150 000	Air	2	7	16	4.29
23-4S	1512-13	2	28	0	0	0	28	148.4	6 360 000	Air	9	47	50	0.66
35-2S Ls	1275-77	3	3	−0.81	−1.16	−25	32	150.8	5 490 000	Air	109	195+	195	0.03
38-2S	1358-59	2	4	−0.12	−0.03	−19	28	149.0	5 230 000	Air	5	112	113	0.28
	1366-67	2	3	−0.28	−0.27	−25	28	148.8	5 170 000	Air	3	151+	151	0.17
40-3S	1201-05	5	3	−0.57	−0.47	−26	28	149.2	5 090 000	Air	4	12	26	2.60
	1407-08	2	7	−0.14	−0.47	−10	28	150.1	5 420 000	Air	5	15	23	3.40
54-1S "A"	1457-58	2	28	0	0	0	28	151.9	5 730 000	Air	100	106+	106	0.10
54-1S "B"	1370-72	3	2	−0.61	−0.47	−25	28	147.2	3 900 000	Water	3	20	29	1.32
	1373-74	2	2	−0.53	−0.58	−26	28	147.5	3 980 000	Air	2	14	17	2.02
	1394	1	4	−0.31	0	−14	29	146.2	3 720 000	Air	161+	161+	161	0.02
	1395	1	4	−0.30	0	−14	29	146.8	3 840 000	Air	5	35+	35	0.44
71-1S	1502-03	2	30	0	0	0	30	150.8	5 730 000	Air	10	41	45	0.92

[a] Compared to value at start of freezing.

---

[5] Tentative Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) (C 215 - 47 T), 1947 Supplement to the A.S.T.M. Book of Standards, Part II, p. 277.

been immersed only prior to incorporation into concrete were frozen partially immersed in water. In each case the concrete was completely immersed during thawing. The interior temperature of the beams frozen in air dropped approximately 30 F. during the first hour, after which the temperature dropped approximately 16 F. per hour.

TABLE XIII.—SUMMARY OF FREEZING-AND-THAWING TEST DATA FOR CONCRETE BEAMS WITH IMMERSED AGGREGATE.

Coarse Aggregate	Beam Numbers	Number of Beams	Initial Tests				Start of Freeze			Type of Freeze	Number of Freezing-and-Thawing Cycles			Average Percentage Loss[a] in Dynamic Modulus per Cycle of Freezing and Thawing
			Age, days	Percentage Difference[a] in Weight	Percentage Difference[a] Unit Weight	Percentage Difference[a] Dynamic Modulus	Age, days	Unit Weight, lb. per cu. ft.	Dynamic Modulus, psi.		10 per cent Loss in Dynamic Modulus	30 per cent Loss in Dynamic Modulus	End of Test	
GOOD FIELD PERFORMANCE														
1-1S	1441–42	2	28	0	0	0	28	153.7	6 630 000	Water	115+		115	0.03
67-2S	1307–08	2	8	−0.20	−0.26	−14	59	153.3	6 260 000	Water	101	161+	161	0.10
	1309	1	8	−0.51	−0.46	−14	130	152.9	6 100 000	Water	194	194+	194	0.05
90-1S	1462–63	2	28	0	0	0	28	151.4	6 000 000	Water	106+		106	0.01
BAD FIELD PERFORMANCE														
9-1S	1112–13	2	61	0	0	0	61	146.8	4 900 000	Air	225+			0.03
	1184–92	9	2	−0.87	−0.78	−34	28	148.1	5 220 000	Air	36+		36	0.14
	1310	1	8	−0.54	−0.67	−19	130	148.8	5 790 000	Water	130	194+	194	0.09
	1311–12	2	8	−0.20	−0.44	−15	59	148.3	5 510 000	Water	140	201+	201	0.05
47-2S	1248–50	3	2	−0.68	−9.74	−38	28	149.0	5 360 000	Water	163	254+	254	0.07
82-1G	1242–44	3	3	−0.62	−0.68	−22	28	148.0	5 520 000	Water	4	42	57	0.69
	1483–84	2	30	0	0	0	30	147.8	5 790 000	Water	10	40	41	0.77
	1520–21	2	28	0	0	0	28	147.7	5 760 000	Water	10	43	48	0.69
UNCLASSIFIED PERFORMANCE														
3-1S "D"	1251–53	3	2	−0.86	−0.61	...	28	146.6	5 090 000	Water	8	26	38	1.57
3-1S "H"	1254–56	3	2	−0.91	−0.27	...	28	147.5	5 020 000	Water	15	31	38	1.14
38-2S	1448–49	2	29	0	0	0	29	149.7	5 930 000	Water	112+		112	0.03
40-3S	1245–47	3	3	−0.96	−0.86	−28	28	150.3	5 780 000	Water	29	49	52	0.70
54-1S "B"	1472–73	2	30	0	0	0	30	146.1	3 680 000	Water	45	106+	106	0.14
71-1S	1478–79	2	30	0	0	0	30	150.5	5 490 000	Water	14	57+	57	0.48

[a] Compared to values at start of freezing.

The temperature at the interior of the beams frozen in water decreased at a slower rate, being 20 F. for the first hour and then approximately 6 F. per hr. During thawing the temperature increased from −8 F. to above 50 F. in 1 hr. for both groups of beams.

Other beams from many of the batches of concrete were tested in flexure under third-point loading after curing. Still others were subjected to alternate wetting-and-drying cycles consisting of 24 hr. in an oven at 130 F. followed by 24 hr. in a water bath at 70 F.

RESULTS OF CONCRETE TESTS AND DISCUSSION

The data on the results of freezing-and-thawing tests of concrete have been divided into three general groups: Mixes in which the coarse aggregate was incorporated in an evacuated and saturated state (Table IX); mixes in which the aggregate was incorporated in the concrete after 24 hr. of immersion (Table X); and data for the concrete in which the coarse aggregate consisted of combinations of materials with good and bad performance records. For each of the concretes in this latter category, one of three different aggregates with bad field performance was combined (in a vacuum-saturated state) with stone from source 67-2S (good field performance). The 67-2S aggregate was used in an immersed state since it had been determined previously that vacuum-saturation of this material caused no deterioration in the concrete that was frozen and thawed. The degree of saturation of the inferior aggregates had been found to be of great importance; these materials were evacuated to accentuate their deleterious effect.

TABLE XIV.—SUMMARY OF FREEZING-AND-THAWING TEST DATA FOR CONCRETE WITH COMBINATIONS OF AGGREGATE.

Percentage Bad Aggregate in Coarse Aggregate	Size of Bad Aggregate	Number of Beams	Initial Tests				Start of Freeze			Number of Freezing and Thawing Cycles			Average Percentage Loss in Dynamic Modulus per Cycle of Freezing and Thawing
			Age, days	Percentage Difference			Age, days	Unit Weight, lb. per cu. ft.	Dynamic Modulus, psi.	10 per cent Loss in Dynamic Modulus	30 per cent Loss in Dynamic Modulus	End of Test	
				Weight	Unit Weight	Dynamic Modulus							
COMBINATIONS OF 35-2S CHERT EVACUATED (BAD PERFORMANCE) WITH 67-2S LIMESTONE IMMERSED (GOOD PERFORMANCE)													
2.5	¾-1 in.	3	2	−0.12	−0.48	−24	28	151.2	5 990 000	48	205	216	0.18
2.5	½-¾ in.	3	4	−0.42	−0.07	−26	28	149.8	5 960 000	55	156	182	0.19
5	¾-1 in.	1	4	−0.43	−0.13	−19	28	151.3	5 770 000	31	85	90	0.67
		1	4	−0.47	...	−18	28	150.9	5 635 000	149	163+	163	0.10
5	½-¾ in.	2	4	−0.37	−0.20	−16	28	152.2	6 060 000	36	119+	119	0.18
5	No. 4—1 in.	3	3	−0.78	−0.93	−24	28	150.9	6 100 000	69	173	186	0.19
		1	4	−0.40	−0.20	−15	28	152.2	5 990 000	129	169+	169	0.15
		2	30	0	0	0	30	151.2	5 850 000	28	77+		0.47
10	No. 4—1 in.	3	3	−0.58	−0.54	−24	28	149.7	6 040 000	31	84	91	0.56
		2	4	−0.39	−0.23	−13	28	151.9	6 180 000	7	129+	129	0.19
15	No. 4—1 in.	3	2	−0.54	−0.57	−26	28	151.1	6 040 000	4	23	25	1.31
25	¾-1 in.	2	4	−0.45	−0.36	−18	28	150.0	5 680 000	4	10	16	2.62
25	½-¾ in.	2	4	−0.39	−0.30	−17	28	150.8	5 840 000	4	10	16	2.66
		2	28	0	0	0	28	151.1	6 140 000	4	16	24	1.85
25	⅜-½ in.	2	4	−0.42	−0.46	−17	28	150.2	5 710 000	3	16	16	1.94
25	No. 4—⅜ in.	2	4	−0.37	−0.40	−21	28	149.7	5 620 000	4	36	58	0.66
25	No. 4—1 in.	3	2	−0.40	−0.63	−42	28	150.6	5 920 000	2	12	18	2.24
COMBINATIONS OF 82-1G (EVACUATED) WITH 67-2S (IMMERSED)													
50	No. 4—½ in.	2	2	−0.54	−0.53	−30	28	149.9	5 580 000	7	49+	53	0.62
		2	10	−0.14	...	−7.5	28	150.7	5 920 000	5	41	52	0.72
50	No. 4—1 in.	2	10	−0.11	−0.17	−6.5	28	150.8	5 980 000	7	50	64	0.54
		2	6	−0.25	−0.23	−10	28	149.7	5 650 000	5	23	37	1.17
75	No. 4—¾ in.	2	2	−0.50	−0.67	−30	28	149.8	5 870 000	4	33	44	0.96
		2	10	−0.12	−0.03	−6.7	28	149.4	5 960 000	8	40	59	0.76
75	No. 4—1 in.	2	10	−0.08	−0.20	−5.5	28	150.4	5 980 000	3	18	33	1.51
		2	6	−0.23	−0.20	−11	28	149.5	5 770 000	2	11	30	2.16
COMBINATIONS OF 9-1S (EVACUATED) WITH 67-2S (IMMERSED)													
25	¾-1 in.	3	1	−0.82	−0.40	−51	28	150.7	5 940 000	56	144	146	0.35
	½-¾ in.	3	1	−0.72	−0.67	−52	28	149.9	5 720 000	30	68	76	0.47
	⅜-½ in.	3	1	−0.65	−0.80	−53	28	151.1	5 750 000	45	157	164	0.26
25	No. 4—1 in.	3	3	−0.28	−0.46	−30	28	151.0	6 000 000	11	46	49	0.71
50	No. 4—1 in.	3	3	−0.35	−0.47	−32	28	149.3	5 510 000	2	8	14	3.93
COMBINATION OF CHERT AND LIMESTONE FROM SOURCE 35-2S (EVACUATED)													
15	No. 4—1 in.	1	2	−0.21	−0.27	−32	7	148.9	4 000 000	14	49	84	0.94
15	No. 4—1 in.	3	2	−0.66	−0.47	−44	28	148.7	5 020 000	6	33	37	0.92

Results of Freezing-and-Thawing Tests:

Results of tests on the beams subjected to freezing and thawing are summarized in Tables XII, XIII, and XIV. For each group of beams the average percentage loss in dynamic modulus per cycle of freezing and thaw-

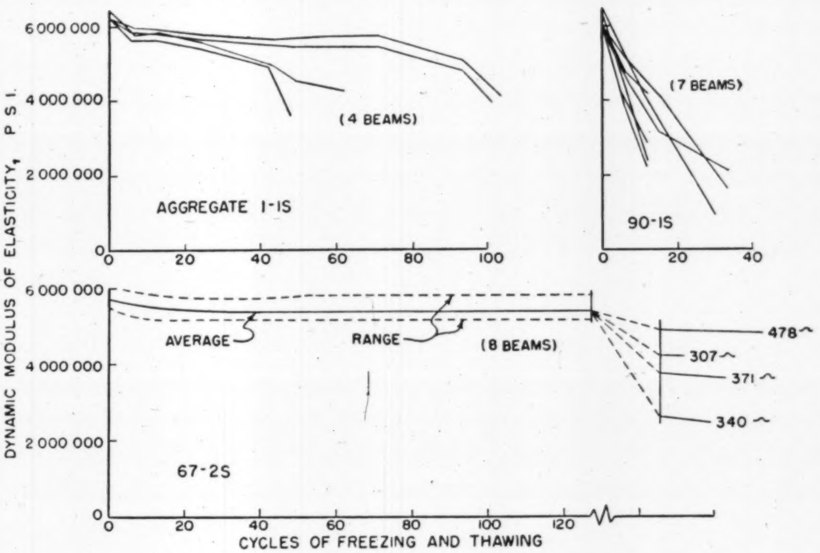


FIG. 1.—Effect of Freezing and Thawing on Dynamic Modulus of Concrete Beams Containing Evacuated Aggregate.

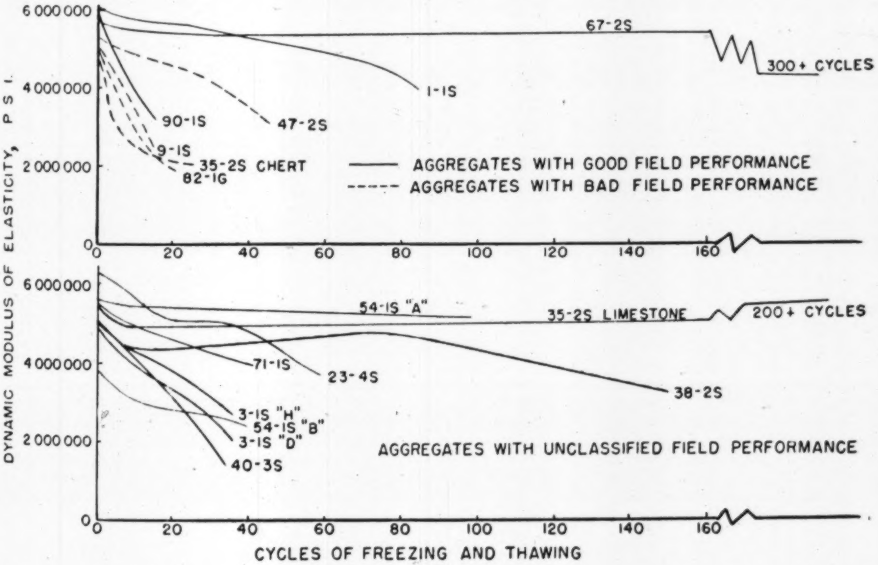


FIG. 2.—Average Dynamic Modulus Curves for Freezing-and-Thawing Tests of Concrete Beams Containing Evacuated Aggregate.

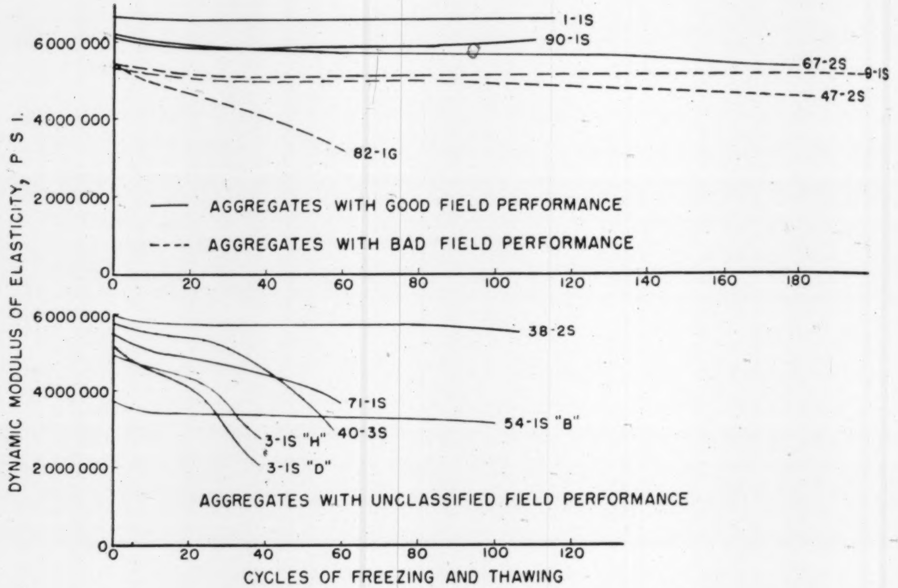


FIG. 3.—Average Dynamic Modulus Curves for Freezing-and-Thawing Tests of Concrete Beams Containing Immersed Aggregate.

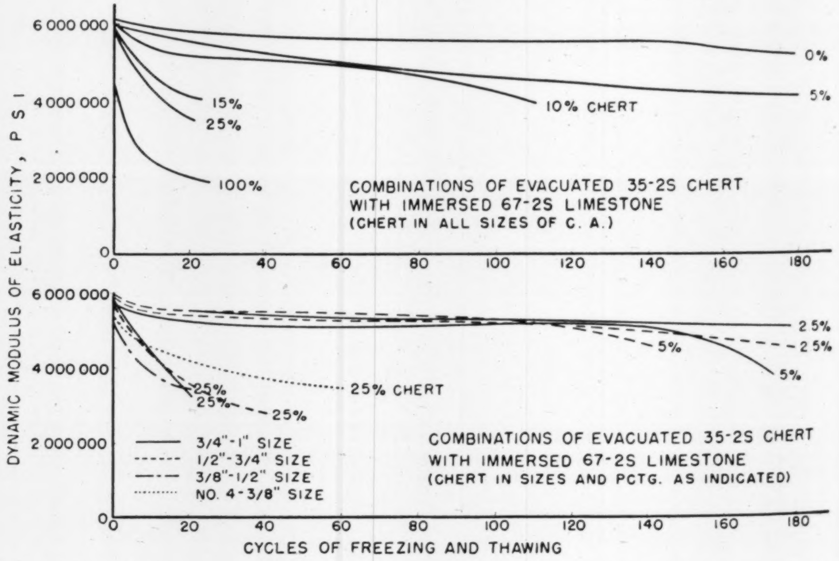


FIG. 4.—Effect of Freezing and Thawing on Dynamic Modulus of Concrete Beams Containing Different Sizes and Percentages of Chert.

ing was calculated as an index of durability. Where the variation between different beams in a single batch was appreciable, the individual values for those beams are tabulated. In the column entitled "Type of Freeze" it is noted whether the beams were frozen in air or partially immersed in water. All of the beams containing combinations of aggregates (Table XIV) were frozen in air. In every case the beams were thawed while totally immersed.

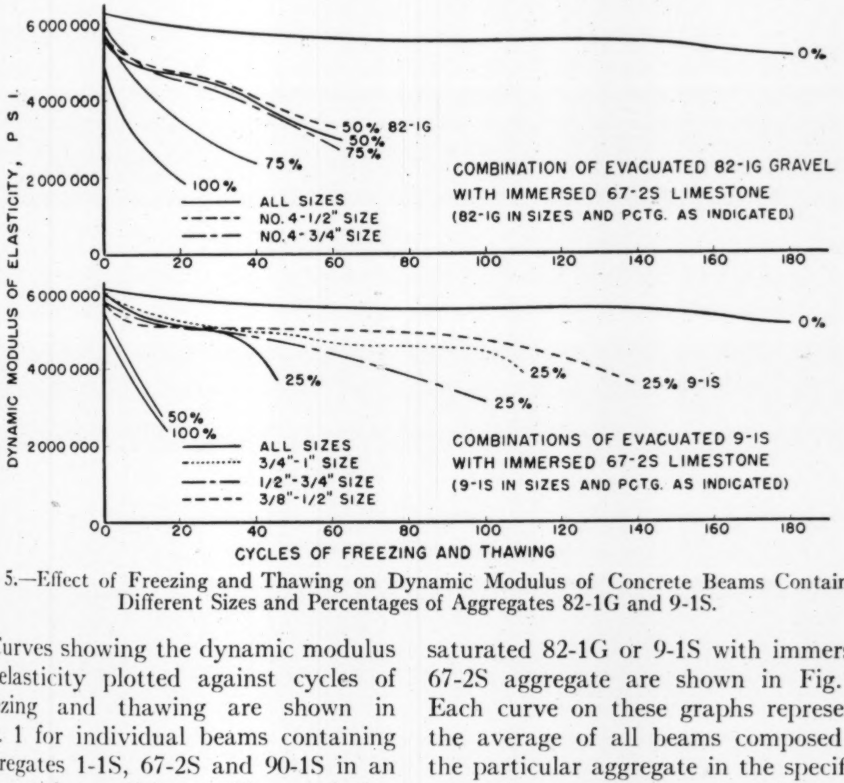


FIG. 5.—Effect of Freezing and Thawing on Dynamic Modulus of Concrete Beams Containing Different Sizes and Percentages of Aggregates 82-1G and 9-1S.

Curves showing the dynamic modulus of elasticity plotted against cycles of freezing and thawing are shown in Fig. 1 for individual beams containing aggregates 1-1S, 67-2S and 90-1S in an evacuated condition. The variation shown between different beams containing the same aggregate is quite typical of the test results. Greater variations than those indicated in Fig. 1 occurred for some aggregates, and for many the uniformity was better. Lack of space prevents the presentation of such curves for all of the individual beams; the data in Tables XII to XIV indicate the extent of variation, and, in addition, the reproducibility of test results is discussed below. Average curves of dynamic modulus *versus* cycles of freezing and thawing are plotted in Fig. 2 for the evacuated aggregates and in Fig. 3 for the concrete containing immersed aggregates. The effect of different percentages of saturated chert is shown graphically in Fig. 4, and curves for combinations of vacuum-saturated 82-1G or 9-1S with immersed 67-2S aggregate are shown in Fig. 5. Each curve on these graphs represents the average of all beams composed of the particular aggregate in the specified saturation condition, and which were moist cured for 28 days prior to the start of freezing and thawing. Data on beams with other curing periods are included in the tables but are not plotted. The performance of the concrete varied from a drop of 30 per cent in dynamic modulus in two cycles of freezing and thawing for the concrete containing aggregate 35-2S chert, evacuated, on one extreme, to the 478 cycles withstood by concrete containing aggregate 67-2S.

Beams containing immersed aggregate were frozen partially immersed in water after it was observed that no change in the weight of the concrete occurred during the process of freezing in air and thawing in water. Up to that time the aggregate tested in an immersed state in concrete had proven highly resistant to the test. The procedure of freezing partially immersed was adopted to give more opportunity for the aggregate to become saturated. However, the data on change in weight and volume indicate that no particular increase in moisture content occurred during the freezing-and-thawing tests. The average change in weight for all beams frozen in air was a loss of 1.0 g. at the time the dynamic modulus had decreased 10 per cent. The average weight change for beams containing immersed aggregate and frozen partially immersed was a gain of 1.1 g. This difference is regarded as negligible. The possibility exists that part of the water absorbed during curing was taken up by the aggregate. However, the average gain in weight for concrete containing evacuated aggregate was 0.82 per cent between 2 and 28 days, and it was 0.80 per cent for the concrete containing immersed aggregate. Because of the variability in gain in weight, this difference is not considered significant.

The variation in resistance to freezing and thawing of different beams containing the same aggregate in the same moisture condition was analyzed in the following manner: The durability index of each beam was divided by the average index for all of the beams made with that aggregate with the particular saturation treatment. After this reduction of all data to a common basis, the standard deviation of the ratios was determined to be 0.49. This value indicates that the reproducibility of the test was such that 67 per cent of the values for durability index fell within a range of plus or minus 50 per cent of the mean value for a particular aggregate. Ninety per cent fell within a range of plus or minus 80 per cent of the mean.

Mortar Saturation:

The data on change in weight of the concrete showed that in every case the concrete increased in weight from the age of 2 days, when the molds were removed, to the age of 28 days. This gain in weight could be interpreted as absorption of water either in the mortar component or in the aggregate component of the concrete. However, the concrete with evacuated aggregate showed gains in weight of the same order of magnitude, and because of the high initial saturation of the aggregate, it was considered unlikely that any further absorption of water by aggregate occurred. Hence, this gain in moisture may be attributed to the absorption of water in the mortar. At the same time the unit weight increased in varying amounts. Part of the increase in unit weight occurred during the first two days, during which time there was no change in weight of the concrete. This change in unit weight must be attributed to shrinkage of the concrete. Further increases in the unit weight would be caused by the additional absorption of water as well as by further shrinkage. Both would have as their effect a decrease in air content. However, an opposite effect is the shrinkage in the sum of the volumes of the cement plus hydrated water components. It was considered desirable to calculate, considering these factors, the air content of the concrete at 28 days in order that an estimate of the degree of saturation of the mortar might be made. Since it has been established firmly that the degree of saturation of materials in general, subjected to freezing action, controls to a large extent their resistance to freezing (**27, 35**) the performance of the concrete beams in this study might be affected by disruption in the mortar as well as by the performance of the aggregate. The mortar saturation, expressed as a ratio of the volume of freezable water to the sum of the volumes of air and freezable water, was calculated by using the relationships between the specific volume of water in hardened cement paste, freezable water content, and "nonevaporable water content" which were developed by Powers and Brownyard (**18, 19, 20**).

It can be shown (**25**) that V'_A, the air volume of hardened concrete per unit of concrete volume, can be calculated from the following formula:

$$V'_A = \frac{\Delta\omega}{\gamma_0} - p + (1 + p)\left(\frac{0.279\ C\omega_n - \Delta\omega}{62.4} + V_{A_0}\right)$$

where:

$\Delta\omega$ = change in water content in pounds per unit volume of concrete,
γ_0 = initial unit weight of concrete (fresh),
p = ratio of change in unit weight to initial unit weight of concrete,
c = weight of cement per unit volume of concrete,
ω_n = ratio of the nonevaporable water content to the weight of cement, a constant which varies with curing and type of cement, and
V_{A_0} = initial air volume per unit volume of concrete.

The weight of water freezable at −20 C. is given by Powers and Brownyard (**19**, p. 965) as

$$\omega_f = \omega_t - 1.96\ \omega_n$$

where:

ω_f = the weight of freezable water, and
ω_t = the total weight of water.

The degree of mortar saturation indicated in Tables IX to XI was calculated as $\dfrac{V\omega_f}{V\omega_f + V'_A}$, where $V\omega_f$ was the volume of freezable water. The air content was determined from a graphical solution of the formula, using an assumed value of 0.23 for ω_n (**17**, p. 249) and determined values for the other variables. The specific volume of the freezable water was taken as 1.0 since Powers and Brownyard state (**20**, p. 992) that for practical purposes the freezable water can be considered as capillary water, the specific volume of which they determined to be 1.0.

The degree of saturation of the mortar, thus calculated, varied for the individual beams from 0.26 to 0.84, averaging 0.63. However, there was no relationship observable between the value for mortar saturation and the resistance of these concretes to freezing and thawing. Hence, it is concluded that the mortar saturation in this range was not critical. Since the value of nonevaporable water content was an assumed one, calculations of mortar saturation also were made using lower values for this factor. Use of a value of ω_n of 0.15 gave indicated mortar saturation values greater than 100 per cent for many of the concrete beams which withstood more than 100 cycles of freezing and thawing with very little deterioration. On the assumption that such a saturation would cause rapid deterioration under freezing action, it is concluded that the first value of ω_n, 0.23, is likely to be the more correct one. However, the use of any particular ω_n in this analysis merely serves to establish the relative mortar saturations, and, as noted previously, the higher values so determined did not appear to be critical.

Further data on the mortar saturation were obtained by evacuating and saturating companion beams to those tested by the regular freezing-and-thawing procedure. The volume of additional water absorbed approximated closely the calculated volume of air. Two of these beams were frozen and thawed after vacuum-saturation, and they lost 30 per cent in dynamic modulus in approximately 20 cycles; other beams from the same batches, tested without saturating the concrete, withstood more than 150 cycles.

Comparison of Aggregate Test Results and Field Performance:

A primary object of this investigation was the determination of methods by which aggregates giving good field performance could be differentiated from those with unsatisfactory field performance in concrete pavements. From Table II it may be seen that general stratigraphic classifications do not serve this purpose. However, the possibility exists that more precise stratigraphic subdivisions will prove consistent in performance over considerable areas and further study should be conducted on this point. Both dolomitic and high-calcium limestones have exhibited both good and bad performance in pavements. All of the limestones tested were relatively "pure," the calcium and magnesium carbonate content varying from 90 to 96 per cent (**25**).

The failure of the sodium sulfate test to reflect field performance is especially serious since this test is used extensively for this purpose. This same observation has been made by a number of investigators. Table VI shows that a correct separation of good and poor performance materials may be made on the basis of the unconfined freezing-and-thawing test with the aggregates in a vacuum-saturated state. However, material 9-1S with a very poor service record showed a loss of only 8.5 per cent, so that a relatively low permissible loss would have to be established as a criterion of durability. The relatively small amount of breakdown for this material indicates that volume change on freezing takes place without rupture of the aggregate particles, since concrete made with it showed rapid deterioration in freezing and thawing.

No relationship between apparent specific gravity, bulk specific gravity, or absorption, and field performance is discernible in Table III. Neither is there complete correlation between service record and total void volume or degree of saturation in an immersed or evacuated state (Table IV). However, the data from the microscopic determination of aggregate void characteristics show that the volume of voids less than 0.005 mm. in diameter for aggregates with good service records was less than 0.06 (expressed as a ratio to solid volume), while it was greater than 0.10 for those with poor records. This relationship also held true for the materials with unclassified records but on which a tentative field rating may be placed. There are several features of interest in connection with this correlation. Common aggregate materials with a percentage absorption of 2 per cent or less when highly saturated would have a total void volume less than 0.06, hence would be expected to show good performance. (Volume of absorbed water = percentage absorption × aggregate true specific gravity.) This checks with a number of studies which have reported a relationship between absorption and durability (**6, 27, 36**). On the other hand, more porous materials may or may not have a large volume of extremely small voids. The significance of the void size in these materials is probably related to their water-retention capacity and capillary action as influencing their degree of saturation. It is not considered that the data reported enable the selection of a precise critical value for this property although it is likely to be between 0.06 and 0.10. The critical void diameter may differ from 0.005 mm., although it is likely to be of this order of magnitude.

Rhoades and Mielenz (**22**, p. 586) stated in this connection, "Water moving by capillarity will not enter aggregate containing only large voids, even if these voids are interconnected and penetrable. On the other hand, small voids will be penetrated; and if these openings are smaller than those of the cement paste, the water will be preferentially drawn into them from the paste. During periods of hydration or drying, while water is being withdrawn from the interstices of cement paste, water will be drawn by capillarity from aggregate particles containing only voids which are larger than those in the paste; but the last residuals of water remaining in even relatively dry concrete may be concentrated in aggregate-voids smaller than those in the cement paste." The arbitrary value of 0.005 mm. was taken as the basis for classification of the pore size by Scott Russell (**23**, p. 34). Voids smaller than this were termed micropores and those larger were termed macropores. A correlation between the weathering properties of building limestones and the volume of micropores was shown by Russell. No limiting value was set; values of microporosity varied from 8.2 to 29.5 per cent of the stone volume.

The relationship between aggregate field performance and the results of the freezing-and-thawing tests of concrete is indicated graphically in Figs. 2 and 3. When the aggregate was incorporated in the concrete in a vacuum-saturated condition, all of the materials with poor records (indicated by the dashed lines in Fig. 2) and material 90-1S with a good record caused decreases of 30 per cent in dynamic modulus in less than 50 cycles of freezing and thawing. When the aggregate was used after being immersed for only 24 hr., the concrete containing the good aggregates withstood more than 100 cycles of freezing and thawing with practically no deterioration. However, in this saturation condition, materials 9-1S and 47-2S with poor field performance also showed no deterioration in more than 100 cycles of freezing and thawing. When the degree of saturation of materials in their natural "quarry-wet" condition is taken into account, it is seen that the results of the concrete freezing-and-thawing test with the aggregate in a condition corresponding to its field moisture content conform without exception to the service records. Materials 90-1S and 1-1S, with good records, showed low field saturations and, in the concrete tests with these aggregates in a 24-hr. immersed condition, the concrete showed high resistance to freezing and thawing. Material 67-2S, with an excellent field record, low degrees of saturation, and absorption less than 1 per cent, showed excellent resistance to freezing and thawing in concrete with the aggregate in either the evacuated or immersed condition. On the basis of the comparison of laboratory test results and field performance records, it is concluded that freezing-and-thawing tests of concrete may be used to differentiate between materials with good and poor field performances.

The field performance data, results of freezing-and-thawing tests on concrete beams, and void volume data are summarized in Table XV. In this tabulation the aggregates are arranged in the order of the resistance to freezing and

TABLE XV.—RELATIONSHIP BETWEEN FIELD PERFORMANCE AND PERTINENT AGGREGATE TEST RESULTS.

Coarse Aggregate	Field Performance	Field Percentage Saturation	Average Test Data in Condition Corresponding to Field Saturation						Volume of Voids <0.005 mm. in Diam.	Average Concrete Freezing-and-Thawing Test Data in Other Saturation Condition			
			Method of Saturation	Percentage Saturation	Concrete Freezing-and-Thawing Tests			Percentage Dynamic Modulus Loss per Cycle of Freezing and Thawing		Method of Saturation	Percentage Saturation	Number of Beams	Percentage Dynamic Modulus Loss per Cycle of Freezing and Thawing
					Number of Beams	Number of Cycles for Dynamic Modulus Loss							
						of 10 per cent	of 30 per cent						
90-1S	Good	66	Immer.	45	2	106+		0.01	0.01	Evac.	87	7	4.18
1-1S	Good	66	Immer.	55	2	115+		0.03	0.05	Evac.	82	4	0.49
38-2S	Unknown	Low	Immer.	59	2	112+		0.03	0.02	Evac.	80	4	0.24
35-2S Ls	Unknown	93	Evac.	77	3	195+		0.03	0.02				
67-2S	Good	33	Immer.	51	3	132	180+	0.08	0.02	Evac.	55	8	0.14
54-1S "A"	Unknown		Evac.	83	2	100	106+	0.10					
23-4S	Unknown		Evac.	87	2	9	47	0.66	0.05				
47-2S	Bad	High	Evac.	95	2	12	36	0.91	0.11	Immer.	71	3	0.07
71-1S	Unknown		Evac.	85	2	10	41	0.92	0.07	Immer.	73	2	0.48
54-1S "B"	Unknown		Evac.	97	7	4	25	1.21	0.08	Immer.	72	2	0.14
3-1S "H"	Suspicious	96	Evac.	90	12	5	17	2.64	0.20	Immer.	81	3	1.14
40-3S	Suspicious	95	Evac.	91	7	4	13	2.84	0.12	Immer.	78	3	0.70
3-1S "D"	Suspicious	97	Evac.	96	11	5	17	2.97	0.14	Immer.	83	3	1.57
9-1S	Bad	94	Evac.	97	17	8	14	3.17	0.12	Immer.	71	3	0.07
82-1G	Bad	100	Evac.	86	6	2	5	6.67		Immer.	76	7	0.71
35-2S Ch	Bad	100	Evac.	98	3	1	2	12.33	0.11				

TABLE XVI.—EFFECT OF DIFFERENT PERCENTAGES AND SIZES OF DELETERIOUS MATERIALS ON RESISTANCE OF CONCRETE TO FREEZING AND THAWING.

Deleterious Material			Number of Beams	Number of Freezing-and-Thawing Cycles to		Percentage Loss Dynamic Modulus per Cycle of Freezing and Thawing
Percentage	Size	Type		10 Per Cent Loss in Dynamic Modulus	30 Per Cent Loss in Dynamic Modulus	
2.5	¾-1 in.	35-2S Chert	3	48	205	0.18
2.5	½-¾ in.	35-2S Chert	3	55	156	0.19
5.0	½-¾ in.	35-2S Chert	2	36	120+	0.18
5.0	No. 4—1 in.	35-2S Chert	6	65	141	0.24
25.0	⅜-½ in.	9-1S	3	45	157	0.29
25.0	¾-1 in.	9-1S	3	56	144	0.35
5.0	¾-1 in.	35-2S Chert	2	90	124	0.38
10.0	No. 4—1 in.	35-2S Chert	5	21	102	0.41
25.0	½-¾ in.	9-1S	3	30	76	0.48
25.0	No. 4—⅜ in.	35-2S Chert	2	5	36	0.66
50.0	No. 4—½ in.	82-1G	4	6	45	0.67
25.0	No. 4—1 in.	9-1S	3	11	46	0.71
50.0	No. 4—1 in.	82-1G	4	6	37	0.86
75.0	No. 4—¾ in.	82-1G	4	6	36	0.89
15.0	No. 4—1 in.	35-2S Chert	3	4	23	1.31
75.0	No. 4—1 in.	82-1G	4	3	15	1.84
25.0	⅜-½ in.	35-2S Chert	2	3	16	1.93
25.0	No. 4—1 in.	35-2S Chert	3	2	12	2.24
25.0	½-¾ in.	35-2S Chert	4	4	13	2.25
25.0	¾-1 in.	35-2S Chert	2	4	10	2.62
50.0	No. 4—1 in.	9-1S	3	2	8	3.93

thawing of the concrete in which they were incorporated, in the saturation state most nearly representative of their field saturation. The materials with good records and three of those upon which no field performance data are available all withstood 100 or more cycles of freezing and thawing without losing more than 10 per cent of their initial dynamic modulus. The percentage loss per cycle for all of these materials was 0.10 or less. All of the materials with unsatisfactory performance records lost 30 per cent of their initial modulus in less than 40 cycles of freezing and thawing, a loss of 0.90 per cent or greater per cycle.

The effect of different percentages and sizes of deleterious material may be evaluated in the light of these data. In the beams with combinations of aggregates, the component with poor field performance was incorporated in a vacuum-saturated condition and was combined with aggregate 67-2S. The data plotted in Figs. 4 and 5 and tabulated in Table XIV are summarized in Table XVI. The different combinations are arranged in this table in the order of the resistance of the concrete to freezing and thawing as indicated by the factor of percentage loss in dynamic modulus per cycle of freezing and thawing. The data are consistent in that the higher the percentage of deleterious material the lower is the resistance to freezing and thawing. By comparison with the aggregates with definite service records, the concrete in which 15 per cent or more of 35-2S chert was incorporated would be classified as unsatisfactory. The concrete containing 75 per cent or more of 82-1G aggregate or more than 25 per cent of 9-1S would also be rated unsatisfactory. On the other extreme, 5 per cent of 35-2S chert did not affect the durability markedly.

Some interesting trends are indicated in Table XVI and Figs. 4 and 5 with respect to the factor of size of deleterious material. The 35-2S chert in a size from No. 4 to $\frac{3}{8}$-in. is markedly better in performance than material larger than the $\frac{3}{8}$-in. sieve, although 25 per cent of the small-size material reduced the durability more than appears to be allowable. Variations in size between $\frac{3}{8}$-in. and 1-in. did not affect the durability appreciably for any of the three materials.

It should be observed that the criterion of performance involving volume of voids smaller than 0.005 mm. in diameter cannot be applied to the average of nonhomogeneous materials. The data on combinations of aggregates show that a relatively small amount of deleterious material may render the aggregate unsuitable even though the weighted average volume of sub-microscopic voids for the aggregate as a whole may be below the critical value.

Further restrictions should be placed on the use of these criteria from the consideration of the field conditions upon which the performance data are based. Some aggregates with unsatisfactory performance under climatic conditions in Indiana might be entirely satisfactory in other regions. By the same token, other factors rendering aggregates unsuitable but having little relationship to void characteristics certainly exist and may also be present in Indiana. It may be possible to develop procedures by which aggregates having the characteristics reported can be used with safety. For example, construction practices have been such that, in many cases, aggregate was incorporated in concrete pavements in a highly saturated state. In view of the excellent performance in the freezing-and-thawing test of material 9-1S when incorporated in the concrete in an immersed state, it is strongly indicated that the aggregate in the concrete pavements which showed severe deterioration did not have an opportunity to dry, since it has also been shown that the drainability of the sub-grade has a strong influence on the performance of the pavements (**36, 30**). It might be concluded that in cuts in plastic soils at least the lower parts of concrete pavements do not become dried and that the concrete undergoes freezing action in a condition analogous to that of the concrete beams in the test procedure used in this study; that is, the concrete was continuously moist cured and no opportunity was allowed for the drying out of either mortar or aggregate.

It is of interest that Cantrill and Campbell reached conclusions regarding criteria for predicting field performance of coarse aggregate which were roughly similar to those discussed above. After studying the durability of Kentucky aggregates they concluded that objectionable aggregate might be eliminated by specifying (**6**):

> "(*a*) Coarse aggregate shall not show an absorption greater than 3 per cent when subjected to A.S.T.M. Standard Test C 95–36.[6]
>
> "(*b*) Concrete in which any aggregate is incorporated shall not show a reduction in flexural strength greater than 30 per cent when subjected to 40 cycles of freezing and thawing in the presence of water."

The 3 per cent limitation in water would, if the aggregate were completely saturated, correspond to approximately 0.08 total volume of voids. However, the presence of many very small voids would tend to lower the saturation so that a material with void space as great as, perhaps, 0.15 might not show greater than 3 per cent absorption. A case of this sort is aggregate 40-3S, which showed an absorption, after 24 hr. of immersion, of 3 per cent but a volume of voids smaller than 0.005 mm. in diameter of 0.100; its total void volume was 0.13.

The limitation of 30 per cent decrease in modulus of rupture would correspond approximately to a 20 per cent decrease in dynamic modulus or a decrease of 0.50 per cent in dynamic modulus per cycle of freezing and thawing. This is in agreement with the test data reported in this investigation although it should be emphasized that the saturation of aggregate and mortar in the laboratory concrete test should correspond to field conditions. Further research is required to evaluate other variables involved in the laboratory freezing and thawing of concrete.

Certain of the aggregates, 9-1S, 3-1S "H", and 40-3S, with poor or suspicious field records and which deteriorated rapidly in the concrete freezing-and-thawing tests were described in Table III as finely laminated dolomites. Material having this laminated character has been consistent, in Indiana, in demonstrating poor performance in concrete pavements. However, not all of the poor-performing materials are laminated. Another difficulty associated with using this appearance as an acceptability criterion is that of definition. It is difficult to specify the difference between laminated stone and stone with closely-spaced bedding planes; the latter may be entirely durable aggregate. McMillan and Ward (**14**) in 1929 cautioned against the use of materials with "... closely spaced bedding lamellae and high insoluble matter, especially if clay minerals can be microscopically detected .... If a high porosity accompanies any of the above, the aggregates should be given close scrutiny."

Change in Length of Concrete Beams and Interpretation of Test Results:

The complete lack of blowups in pavements without expansion joints constructed with the good-performing aggregates such as 67-2S indicates a significant departure from the commonly-accepted theory which has led to the widespread incorporation of expansion joints in pavements. On the other hand, as many as 25 blowups per mile have occurred in the pavements constructed with aggregate 9-1S. The differences indicated between pavements containing these aggregates might be due to higher coefficient of thermal expansion for concrete containing 9-1S, greater expansion due to increase in moisture content, lower resistance to buckling action, residual growth due to weathering action or chemical reaction, or combinations of these. Lu (**13**) determined the coefficient of expansion of concrete containing aggregates 67-2S, 9-1S, and 82-1G and found values, in a saturated state, of 0.0000059, 0.0000093, and 0.0000100 in. per inch per degree Centigrade, respectively. Change in length due to change in moisture content was approximately the same for the three materials. Thus the difference in blowup performance might be due to differences in thermal expansion of the concrete. (These coefficient of expansion values for the concrete were close to the coefficients of the aggregates themselves.)

---

[6] Standard Method of Field Test for Absorption of Mining Water by Aggregates for Concrete (C 96 - 36), 1936 Book of A.S.T.M. Standards, Part II, p. 332. (This Standard has been replaced by the Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregat (C 127 - 42), 1946 Book of A.S.T.M. Standards, Part II, p. 470.)

The tendency of the concrete to gain in length during the freezing-and-thawing tests is of interest with respect to residual growth. The data on change in length of the concrete beams were more erratic than were the other measurements made, probably because of lack of control of the temperature of the beams at the time the length tests were made. However, there was a consistent growth in length shown as the concrete deteriorated. The average values for change in length at 10 per cent loss in dynamic modulus and 30 per cent loss in dynamic modulus were, respectively, 0.018 per cent and 0.050 per cent increase in length. It is suggested that the deterioration of the concrete with accompanying growth in length takes place under freezing-and-thawing conditions in the winter time. Added to this permanent growth is the tendency toward lengthening caused by high moisture contents in the spring of the year and, finally, the expansion due to temperature rises, so that the total expansive force exceeds the resistance of the slab to the buckling action, and a blowup occurs. That such residual growth can occur because of other factors is recognized and is probably of importance in some cases in Indiana. Lu (**13**) showed that a heating-and-cooling cycle in which concrete beams were alternated from a hot water bath at 130 F. to an ice water bath at 32 to 38 F. would cause rapid deterioration and gain in length of concrete containing aggregate 82-1G in only ten cycles. It is indicated that concrete in service might be similarly affected by alternations of temperature without freezing action, although the great disparity between rate of temperature change in Lu's tests and field conditions needs to be evaluated. Since alternations in temperature had this effect, it should be recognized that freezing-and-thawing tests may combine this sort of action with the forces of ice crystallization. It may be the reason for the relatively rapid deterioration of the concrete beams containing aggregate 82-1G in an immersed state as shown in Table XIII.

Growth in length and map-cracking of concrete from a test consisting of 24 hr. in water at 70 F. followed by 24 hr. in a drying oven at 130 F. was reported by Gibson (**8**) and Jackson and Kellerman (**11**). Concrete beams containing aggregates 67-2S, 9-1S, 35-2S, 47-2S, 82-1G, 3-1S, and 40-3S have been subjected to this treatment. No cracking or consistent length change has occurred in approximately 150 cycles.

RELATIONSHIP OF MODULUS OF RUPTURE AND DYNAMIC MODULUS OF ELASTICITY

The change in dynamic modulus of elasticity has been used to a considerable extent in concrete durability studies to predict the change in modulus of rupture. Reagel (**21**) showed that the relationship between these factors was indicated by the formula:

$$R = 6\,E^{0.6}$$

where R was percentage reduction in modulus of rupture and E was percentage change in dynamic modulus. Analysis of data in this investigation indicated that an equation of this form was most suitable. The best fitting line by least squares methods was found to have the equation $R = 5.2\,E^{0.65}$.

It was observed that the flexural strengths of the concrete beams kept continuously moist were directly related to the dynamic moduli of elasticity. After analyzing this relationship for each aggregate separately, it was determined that all of the concrete made with 12 of the 16 aggregates could be grouped in considering the correlation between these two properties. For all of the beams made with these aggregates, the following relationship applied for values of dynamic modulus above 3,000,000 psi.:

$$y = 0.00018\,x - 335$$

where y is the flexural strength in psi., and x is the dynamic modulus of elasticity in psi. The correlation coefficient for the data in this range was 0.95 and the standard error of estimate was 61 psi.

The linear character of the relationship did not hold below dynamic modulus values of 3,000,000 psi. The gravel, 82-1G, showed markedly higher modulus of elasticity for a given strength. Three of the crushed stone aggregates (and also beams composed of sand-cement mortar) showed appreciably lower modulus of elasticity for a given strength. Munger (**16**) also showed that the relationship of modulus of elasticity and modulus of rupture was approximately linear but differed for different aggregates.

CONCLUSIONS

1. As a result of this investigation, it is concluded that Indiana coarse aggregates with poor field performance can be differentiated from those with good performance by freezing-and-thawing tests of concrete beams containing the aggregates. Materials with good field performance withstood more than 100 cycles of freezing and thawing in concrete with less than 10 per cent loss in dynamic modulus of elasticity (equivalent to approximately 20 per cent loss in flexural strength). Those with poor field records lost 30 per cent in dynamic modulus (approximately 50 per cent in flexural strength) in less than 40 cycles of freezing and thawing.

It is indicated further that Indiana aggregates without established field records can be evaluated on the basis of freezing-and-thawing tests on concrete. In such tests the aggregate should be incorporated in the concrete in a vacuum-saturated condition with the exception of materials with a ratio of less than 0.06 between the volume of pores smaller than 0.005 mm. in diameter and the volume of solids. In the latter instance, the aggregates should be tested at a lower degree of saturation than that obtained by evacuation unless their moisture contents in the quarry-wet state correspond to those obtained by vacuum saturation.

Furthermore, the mortar in the concrete should have a low degree of saturation. Because the influence of other variables, such as the type of freezing-and-thawing test and the character of the cement, is imperfectly understood, the tests should be set up on a comparative basis, comparing the laboratory results on beams containing the aggregate in question to results of tests with other materials with established field performance.

2. The volume of pores smaller than 0.005 mm. in diameter appears to be a critical index of field durability of Indiana aggregates, probably because of the influence of pore size on water-retention and capillary characteristics of the rock material. Materials having a volume of voids smaller than 0.005 mm. of less than 0.06 (per unit of solid volume) had good service records in concrete pavements, while those having a value of this criterion of 0.10 or greater had demonstrated unsatisfactory performance in pavements.

3. The growth in length of the laboratory-made concrete beams containing aggregates which caused excessive blowing up of pavement concrete, and the occurrence of cracking and loss of strength in these concrete beams in freezing-and-thawing tests, suggests that the analogous deterioration of pavement concrete is caused by freezing and thawing action, followed by normal expansion due to increase in temperature and moisture content resulting in pavement blowups. It has been demonstrated in other studies that permanent length and volume increase can also occur from alkali-aggregate reaction, alternate wetting and drying, and alternations of temperature without freezing. The data reported here do not exclude the possibility that one or all of these factors may affect concrete performance in Indiana in some cases; however, investigations of the first two factors have shown negative results to date. Laboratory studies of the effect of alternations of temperature on concrete containing Indiana aggregate showed that deterioration of concrete in the field might be caused by this action in some cases. The occurrence in regions with little freezing action of deterioration of concrete pavements containing aggregates similar to some of those in Indiana also indicates that this action may be a contributing factor.

4. General geological classifications had no generally consistent relationship to the field performance of the aggregates investigated. However, certain formations of limestone were consistent in performance over wide areas. Both dolomitic and high-calcium limestones were included in the materials observed with good fields records and also in those with unsatisfactory performance. The limestones tested in the laboratory all contained between 90 and 96 per cent calcium and magnesium carbonate content, there being no observable relationship, in this range, between field performance and quantity of "impurities."

5. The characteristics of apparent specific gravity, bulk specific gravity, absorption, and total void volume showed no generally consistent relationship to field performance. However, the limestone aggregates with greater than 3.9 per cent absorption and greater than 0.86 degree of saturation when incorporated in concrete in laboratory mixes caused rapid deterioration of the concrete in freezing and thawing. Concrete containing aggregates with an absorption between 2 and 3 per cent and greater than 0.82 deg. of saturation was intermediate in resistance to freezing and thawing. Concrete containing aggregate with a degree of saturation less than 0.8 or an absorption less than 2 per cent was highly resistant to freezing and thawing.

6. The sodium sulfate and unconfined freezing-and-thawing tests did not indicate the field performance characteristics of the aggregates, although unconfined freezing-and-thawing tests of the aggregate in a vacuum-saturated condi-

tion did afford a separation of aggregates with good and poor field performance. The relatively small amount of breakdown when frozen unconfined of some of the aggregates which showed poor performance in freezing-and-thawing tests of concrete indicates that these materials cause concrete deterioration by volume change without rupture of the aggregates themselves.

7. The degree of saturation of the mortar in the concrete made for these tests was low enough that the resistance of the concrete to freezing and thawing was not affected measurably by this factor; the performance of the concrete was controlled by the coarse aggregate. It was shown that the change in air content of the mortar component from the fresh condition to the hardened condition could be determined by considering the change in total water content, the change in unit weight, and the nonevaporable water content.

8. It is concluded, partially on the basis of previously published data and also from the test results reported here, that some of the unsatisfactory aggregates may be used in pavement concrete (where economic conditions are such that this is desirable) if certain precautions are taken. These would include partial drying of the aggregate prior to incorporation in concrete, use of air-entrainment, and use of freely-draining base courses. Additional research is required to establish firmly the limitations of such procedures.

9. Further research is needed to evaluate variables affecting aggregate performance in concrete among which the following topics are suggested:

(*a*) The action of mortar in normal and air-entrained concrete in preventing dried or partially dried aggregate from becoming highly saturated under field or laboratory conditions. Test results in this investigation indicate that, with concrete of the character used, the mortar is highly efficient in this respect, but it is considered that further data are required.

(*b*) The fatigue, elastic, and extensibility characteristics of normal and air-entrained mortar as influencing the damage to the concrete caused by aggregate volume changes.

(*c*) The effect of rate of temperature change, as a factor influencing the comparison of laboratory and field weathering. This applies to both the factors of temperature change without freezing, and freezing-and-thawing action.

Acknowledgments:

The author wishes to express his appreciation to Prof. K. B. Woods, Associate Director of the Joint Highway Research Project, for his valuable guidance and encouragement and review of the manuscript, and to Mr. D. W. Lewis, Research Engineer, Joint Highway Research Project, who aided in planning and conducting the tests. Mr. P. D. Meisenhelder, Engineer of Tests, and Mr. S. W. Benham, Research Engineer, State Highway Commission of Indiana contributed valuable suggestions and reviewed the manuscript. A number of part-time student assistants aided in the test work and their assistance is gratefully acknowledged.

REFERENCES

(1) D. A. Abrams, "Influence of Aggregates on the Durability of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 172 (1923).

(2) A.S.T.M. Standards on Concrete and Concrete Aggregates, Am. Soc. Testing Mats., February, 1947. (Issued as separate publication.)

(3) E. O. Axon, T. F. Willis, and F. W. Reagel, "Effect of Air-Entrapping Portland Cement on the Resistance to Freezing and Thawing of Concrete Containing Inferior Coarse Ag-

gregate," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 981 (1943).

(4) Fred F. Bartel, "Effect of Aggregate Characteristics on Durability of Concrete," *Thesis*, University of Maryland (1942).

(5) S. L. Bugg, "Effect of Air-Entrainment on the Durability Characteristics of Coarse Aggregates," *Thesis* (submitted to Purdue University in partial fulfillment of the requirements for the Degree of Master of Science in Civil Engineering), February, 1948.

(6) Curtis Cantrill and Louis Campbell, "Selection of Aggregates for Concrete Pavement Based on Service Records," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 937 (1939).

(7) A. Casagrande and R. E. Fadum, "Notes on Soil Testing for Engineering Purposes," *Soil Mechanics Series*, No. 8, Harvard University, January, 1940.

(8) W. E. Gibson, "A Study of Map Cracking in Sand Gravel Pavements," *Proceedings*, Highway Research Board, Vol. 10, p. 227 (1938).

(8) F. B. Hornibrook, Howard Freiberger, and Albert Litvin, "A Study of Durability and Void Characteristics of Concretes Containing Admixtures, Principally of the Air-Entraining Type," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1320 (1946).

(10) F. H. Jackson, "Relation Between Durability of Concrete and Durability of Aggregates," *Proceedings*, Highway Research Board, Vol. 10, p. 101 (1930).

(11) F. H. Jackson and W. F. Kellerman, "Volume Changes in Sand-Gravel Concrete," *Proceedings*, Highway Research Board, Vol. 22, p. 252 (1942).

(12) F. C. Lang and C. A. Hughes, "Discussion on Relation Between Durability of Concrete and Durability of Aggregates," *Proceedings*, Highway Research Board, Vol. 10, p. 116 (1930).

(13) N. Y. Lu, "Comparison of the Thermal and Moisture Expansion of Concrete," *Thesis*, (submitted to Purdue University in partial fulfillment of the requirements for the Degree of Master of Science in Civil Engineering), February, 1948.

(14) F. R. McMillan and G. W. Ward, "Influence of Coarse Aggregate on the Durability of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 816 (1929).

(15) R. A. Moyer, "An Eleven-year Study of the Expansion and Contraction of a Section of Concrete Pavement," *Proceedings*, Highway Research Board, Vol. 25, p. 71 (1945).

(16) H. H. Munger, "The Influence of the Durability of Aggregate upon the Durability of the Resulting Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 787 (1942).

(17) T. C. Powers, "A Working Hypothesis for Further Studies of Frost Resistance of Concrete," *Journal*, Am. Concrete Inst., Vol. 16, No. 4, February, 1945 p. 245.

(18) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste, Part 5," *Journal*, Am. Concrete Inst., Vol. 18, No. 6, February, 1947, p. 669.

(19) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste, Part 8," *Journal*, Am. Concrete Inst., Vol. 18, No. 8, April, 1947, p. 933.

(20) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste, Part 9," *Journal*, Am. Concrete Inst., Vol. 18, No. 8, April, 1947, p. 971.

(21) F. V. Reagel, "Freezing and Thawing Tests of Concrete," *Proceedings*, Highway Research Board, Vol. 20, p. 587 (1940).

(22) Roger Rhoades and R. C. Mielenz, "Petrography of Concrete Aggregate," *Journal*, Am. Concrete Inst., Vol. 17, No. 6, June, 1946, p. 581.

(23) R. J. Schaffer, "The Weathering of Natural Building Stones," *Building Research Special Report No. 18*, Dept. of Scientific and Industrial Research, London (1932).

(24) C. H. Scholer, "Some Accelerated Freezing and Thawing Tests on Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 472 (1928).

(25) Harold S. Sweet, "Concrete Durability as Affected by Coarse Aggregates," *Thesis*, (submitted to Purdue University in partial fulfillment of the requirements for the Degree of Doctor of Philosophy), June, 1948.

(26) Harold S. Sweet and K. B. Woods, "Map-cracking in Concrete Pavements as Influenced by Soil Textures," *Proceedings*, Highway Research Board, Vol. 26, p. 286, (1946).

(27) Harold S. Sweet and K. B. Woods, "A Study of Chert as a Deleterious Constituent in Aggregates," *Engineering Bulletin of Purdue University*, Research Series No. 86, September, 1942.

(28) George J. Verbeck, "The Camera Lucida Method for Measuring Air Voids in Hardened Concrete," *Journal*, Am. Concrete Inst., Vol. 18, No. 9, May, 1947, p. 1039.

(29) Stanton Walker, "Soundness of Aggregates," Report on Significance of Tests of Concrete and Concrete Aggregates, Second Edition, Am. Soc. Testing Mats., p. 112 (1943). Issued as separate publication, Technical Publication No. 22(a).)

(30) Stanton Walker, "Discussion on Freezing and Thawing of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 996 (1943).

(31) Stanton Walker, "Freezing and Thawing Tests of Concrete Made with Different Aggregates," *Journal*, Am. Concrete Inst., Vol. 15, No. 6, June 1944, p. 573.

(32) L. V. White and R. L. Peyton, "Condition of Concrete Pavements in Kansas as Affected by Coarse Aggregate," *Proceedings*, Highway Research Board, Vol. 25, p. 129 (1945).

(33) M. O. Withey, "Considerations Involved in the Making of Freezing-and-Thawing Tests on Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1198 (1946).

(34) K. B. Woods, H. S. Sweet, and T. E. Shelburne, "Pavement Blowups Correlated with Source of Coarse Aggregate," *Proceedings*, Highway Research Board, Vol. 25, p. 147 (1945).

(35) F. N. Wray and H. H. Lichtefeld, "The Influence of Test Methods on Moisture Absorption and Resistance of Coarse Aggregate to Freezing and Thawing," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1007 (1940).

(36) C. E. Wuerpel and E. P. Rexford, "The Soundness of Chert as Measured by Bulk Specific Gravity and Absorption," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1021 (1940).

DISCUSSION

Mr. P. D. Miesenhelder[1] (*presented in written form*).—Mr. Sweet's paper is a noteworthy contribution. As long as concrete has been used as a material of construction, engineers have been concerned with a suitable test to determine the quality of coarse aggregate of unknown service record. Specification wordings have ranged from "the particles shall be clean, hard, and durable" to a quantitative figure computed from the results of some so-called "soundness" test. None of these tests has been entirely satisfactory.

In his conclusions 1, 2, and 5 the author describes three laboratory procedures for testing an unknown coarse aggregate to determine whether or not it will produce durable concrete. The simplest of these is the determination of absorption and degree of saturation (conclusion 5). Probably the most reliable procedure advocated by Mr. Sweet is the freezing-and-thawing test of concrete under certain conditions containing the coarse aggregate in question conducted simultaneously with the same test on concrete containing coarse aggregate of satisfactory service record. However, if further research demonstrates the infallability of "Concrete containing aggregate with a degree of saturation less than 0.8 or an absorption less than 2 per cent was highly resistant to freezing and thawing" the problem of identifying suitable coarse aggregate will be simplified tremendously.

Mr. L. E. Gregg.[2]—I should like to ask Mr. Sweet about the source of fine aggregate which was used in these tests—whether the fine aggregate was the same as that which went into the pavement with the coarse aggregates so many years previous to the time of which he speaks now? I will give the reason for that question: He has recommended a comparison between a coarse aggregate of known good quality and any aggregate in question. We made some tests in our laboratory recently in which we were comparing a material, with which we had had no experience whatsoever, and making this comparison with a coarse aggregate which we considered to be of excellent quality. However, in making the comparison we used a fine aggregate from a location very close to that of the new source being investigated. We used that fine aggregate with both types of coarse aggregate.

As it turned out, the performance in laboratory freezing-and-thawing tests with our coarse aggregate of supposedly good quality was very poor. In fact, the failure occurred in perhaps one-fifth of the time we would ordinarily anticipate. In order to investigate this further we are now running both coarse aggregates with a variety of fine aggregates.

I present that question to Mr. Sweet in case he may be able to give us some information that will help us on the question.

Mr. Bryant Mather.[3]—The points that Mr. Gregg has raised follow closely the experience in our laboratory, in that the durability in freezing and thawing of concrete specimens appears to be a function of the *combination* of fine and coarse aggregates used. All other things being equal, if the failure is due to what has commonly been called physical unsoundness, physical unsoundness of the coarse aggregate is a more important factor than similar unsoundness in the fine aggregate. However, with relatively sound materials a wide range in freezing-and-thawing durability has been observed as a function of the combination of relatively sound fine and coarse aggregates.

---

[1] Engineer of Materials and Tests, Indiana State Highway Commission, Indianapolis, Ind.

[2] Associate Director of Research, Kentucky Department of Highways, Highway Materials Research Lab., Lexington, Ky.

[3] Corps of Engineers, Waterways Experiment Station, Mississippi River Commission, Clinton, Miss.

One other point: Mr. Sweet referred to the cracking observed in certain specimens due to temperature cycles above the freezing point. I wonder if some of that behavior may be a function of the phenomenon which he mentioned at the beginning of his paper, of differential expansion between the coarse aggregate and the mortar?

I would suggest further that such behavior might be related to the differences in thermal coefficient of expansion inherent on the one hand in the coarse aggregate, and controlled in the case of the mortar by the type of fine aggregate used in that mortar. If those differences are excessive, stresses may be set up at the interface between the coarse aggregate and mortar which would be sufficient to cause cracking, reduction in modulus of elasticity, and failure of concrete.

MR. A. T. GOLDBECK.[4]—I wish to congratulate Mr. Sweet on the thoroughness of his paper. I think he has brought out a number of very important points regarding some of the causes of lack of durability of concrete.

This matter of the effect of temperature changes was investigated in our laboratory some 10 years ago. We alternated the temperature of the concrete rapidly between 70 and 140 F., and we observed that with each alternation the concrete grew in length to a different amount with different aggregates. Finally, some of the concretes, those showing the highest expansion, exhibited severe cracking on the surface. The reason for this behavior is evident. Sufficient contraction of the outer surface was produced by a lowering of the temperature to create a tensile stress in excess of the tensile resistance of the concrete.

I am delighted to have this paper presented because I think it points the way toward several investigations that should be made when we are examining the causes of concrete disintegration.

MR. H. S. SWEET (*author's closure*).—There have been two or three points raised on which I would like to comment. Regarding the sand used in these tests, it was a material which had been combined in pavement concrete with two of the coarse aggregates investigated; the remainder of the aggregates had been used in pavements with a different fine aggregate.

The question of the effect of the fine aggregate is one that we have not gone into, and I am very much interested in Mr. Gregg's comments on that.

We do have field records which showed rather conclusively that the fine aggregate used with these coarse aggregates in the field was not a contributing factor to the durability of the pavements. That is the strongest evidence we have for feeling that the coarse aggregate performance should be shown in the laboratory with fine aggregate as a very minor factor, because of the lack of correlation between fine aggregate and durability in the field.

I would concur with Mr. Mather and Mr. Goldbeck about the factor of differential thermal properties between the aggregate and the mortar being the probable cause for cracking in the tests using temperature alternations above freezing. That particular series of tests was not a part of the scope of this paper but was referred to because the tests were conducted with the same aggregates. The occurrence of cracking in laboratory concrete, under these conditions, suggests that concrete in the field may be affected similarly by temperature alternations without freezing action.

---

[4] Engineering Director, National Crushed Stone Assn., Inc., Washington, D. C.

FACTORS IN THE RESISTANCE OF BRICK MASONRY WALLS TO MOISTURE PENETRATION*

By C. C. Connor[1]

Synopsis

The materials and practices used at ninety-one brick-masonry buildings were studied. Roofs or parapet wall coverings, high lime content mortars, moderate rate of absorption bricks, and concave mortar joint tooling were indicated to be factors highly favorable to the moisture resistance of the walls; high water retentivity in mortar, and wall furring were desirable. It was indicated that a combination of the four highly favorable factors with none of the detrimental factors was required to consistently produce moistureproof buildings. Spandrel beam waterproofing, parapet flashings that did not completely cut off or cover the top and back of parapets, high or low rate of absorption bricks, low lime content mortars, and flat tooled, rough cut, and raked mortar joints were indicated to be detrimental to the moisture resistance of the walls. The kind and type of lime used in the mortar, the type and thickness of the walls, the back-up materials, the height of the buildings, and the molding process of the brick, exerted no significant influence on moisture resistance. The results obtained at forty-three buildings, designed to incorporate the materials and practices indicated as most desirable for moisture resistance by laboratory research, field studies and experience, are reported.

Moisture penetration through walls above grade has been one of the most serious problems in brick masonry construction. The purpose of this investigation was to study the materials and practices used in existing brick buildings exposed to the weather for considerable periods, and, if possible, to identify the following:

1. The factors that were favorable or unfavorable to the moisture resistance of brick walls above grade.

2. The materials and practices which exerted no influence on the moisture resistance of walls.

3. The combination of factors required to consistently produce moistureproof brick masonry walls above grade.

This investigation is one of a series of field studies. The first was a study of existing buildings to determine the causes of leaks. The second was on the amount of cracking that occurred between mortar joints and bricks at existing buildings, the results of which were presented before this Society in 1934 (**1**).[2] A third investigation was a survey of the results obtained from procedures used to stop leaks at buildings in a long-term moistureproofing program.

In 1937 a building was designed to incorporate the materials and practices which, based upon the information available at that time from laboratory investigations (**2**, **3**, **4**, **5**, **6**, **7**), field studies, construction experience, and results obtained in moistureproofing work, gave the most promise of stopping the water at the face of the wall and thus prevent its penetration. That building proved moistureproof. It was followed by a long series of others designed on the same principles but modified to fit conditions. This study includes forty-three of those buildings.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] New Jersey Bell Telephone Co., Newark, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1039.

Definition of Terms:

Since frequent use is made of a number of terms in this paper, the following definitions or explanations are set forth:

Integral Wall.—As used in this paper, any wall that is not divided into parts. Any wall that is not a cavity or hollow wall.

Separation Cracking or Mortar Joint Cracking.—The terms "separation cracking" or "mortar joint cracking" as used in this paper refer to a condition of lack of adhesion between brick and mortar which is visible at the exterior face of the brickwork, without regard for the cause.

High-Lime Mortar.—As used in this paper, mortars containing an amount of lime equal to or exceeding 50 per cent of the volume of the cement.

Low-Lime Mortar.—As used in this paper, mortars having a lime content less that 50 per cent of the cement by volume.

High Water Retentivity.—As used in this paper, the ability of a mortar to retain water and thus produce a flow after suction of 65 per cent or more of the initial flow as determined by methods conforming to those used for testing masonry cements.[3]

Low Water Retentivity.—As used in this paper, the ability of a mortar to retain water and thus produce a flow after suction of less than 65 per cent of the initial flow as determined by methods conforming to those used for testing masonry cements.[3]

Specially Hydrated Lime or Special Hydrate.—As used in this paper, hydrated limes which are differentiated from normally hydrated limes or normal hydrates by their ability to develop higher water retentivity in mortar, and in other ways as described for type S and type N limes in the specifications for masonry lime.[4]

BUILDINGS STUDIED

Ninety-one existing buildings were studied. Sixty-seven of these were complete structures, and 24 were original or added sections. Nine of the buildings had walls of different face bricks. These were considered as two units each. Based on the face brick, the study covered a total of one hundred units. The buildings included all of the brick masonry structures built for one company during a 17-yr. period, and their ages varied from 6 to 23 yr. They were all exposed to the severe storms of a seacoast state. The units included a wide scope of materials and practices. Nine different mortars had been used, and 55 makes of brick. The records available for each building were full and complete and were supplemented by field inspections.

The mortar joints had been specified to be filled at all units, but this was not always perfectly done. However, the workmanship was probably considerably better than average. The inspection, in general, was good though periodic in most cases. All units were constructed under commercial conditions, and all but two were built under lump sum contracts awarded to the lowest bidders of a group of selected contractors.

---

[3] Standard Specifications for Masonry Cement (C 91 - 40), 1946 Book of A.S.T.M. Standards, Part II, p. 7.

[4] Tentative Specification for Hydrated Lime for Masonry Purposes (C 207 - 46 T), 1946 Book of A.S.T.M. Standards, Part II, p. 1308.

TABLE I.—TYPES OF BRICK STUDIED AND THEIR CLASSIFICATION BY RATE OF ABSORPTION.

Building Unit	Brick Number	Molding Process[a]	Rate of Absorption, g. Absorbed in 1 min. or ml. Absorbed in 2 min.											
			Low			Moderate			High					
			Chipped Face Average Under 0.03 ml.	Unchipped Face Average Under 0.03 ml.	Average Under 5 g.	Chipped Face Average 0.03 to 0.20 ml.	Unchipped Face Average 0.03 to 0.31 ml.	Average, 5 to 25 g.	Chipped Face Average Over 0.20 ml.	Unchipped Face Average Over 0.31 ml.	Maximum, ml.	Minimum, ml.	Maximum, g.	Minimum, g.
No. 89	1	SC	0.01								0.01	0.005		
No. 58	2	SC	0.01								0.015	0.01		
No. 67	3	SC	0.02								0.03	0.02		
No. 64	4	SC	0.02								0.03	0.02		
No. 90	5[b]	SC	0.02								0.03	0.01		
No. 46	6	SM		0.01							0.015	0.01		
No. 69	7A	SM		0.02							0.02	0.015		
No. 68	7B	SM		0.02							0.02	0.02		
No. 71	7C	SM		0.02							0.04	0.01		
No. 70	7D	SM		0.02							0.03	0.02		
No. 8	8	SC			3.8								6.3	2.6
No. 65A	9	SC				0.05					0.08	0.01		
No. 43A	10	SC				0.06					0.08	0.04		
No. 54	11	SC				0.06					0.06	0.06		
No. 51	12	SC				0.06					0.08	0.04		
No. 73	13	SC				0.07					0.12	0.03		
No. 45	14A	SC				0.08					0.09	0.08		
No. 59	14B	SC				0.08					0.09	0.07		
No. 49	15	SM				0.08					0.10	0.05		
No. 72	16	SC				0.08					0.20	0.01		
No. 75	17	SC				0.08					0.10	0.05		
No. 74	18	SC				0.09					0.11	0.07		
No. 48	19	SC				0.12					0.28	0.00		
No. 47	20	SC				0.19					0.42	0.06		
No. 53A	21	SC				0.20					0.25	0.14		
No. 44	22	SM					0.05				0.10	0.02		
No. 76	23	SM					0.09				0.21	0.01		
No. 6A	24A	SC					0.11				0.21	0.03		
No. 77	25A	SM					0.12				0.19	0.06		
No. 78	25B	SM					0.12				0.20	0.03		
No. 43B	26[c]	SM					0.14				0.33	0.06		
No. 9	24B	SC					0.14				0.40	0.03		
No. 79	28A	SM					0.14				0.25	0.02		
No. 81	29	SM					0.15				0.19	0.08		
No. 80	28B	SM					0.19				0.28	0.08		
No. 82	30A	SM					0.22				0.35	0.08		
No. 52	28C	SM					0.22				0.34	0.05		
No. 86	30B	SM					0.28				0.39	0.24		
No. 10B	31A	SC						5.2					8.6	2.9
No. 3	32	SM						5.8					8.6	2.2
No. 6B	31B	SC						7.5					8.2	7.1
No. 13	33	SM						7.7					15.0	2.5
No. 2	34A	SM						9.5					13.5	6.7
Nos. 14–40	34B	SM						9.9[d]					23.9	1.0
No. 66	34C	SM						9.9[e]					23.9	1.0
No. 11	34D	SM						9.9[e]					23.9	1.0
No. 12	34E	SM						9.9[e]					23.9	1.0
No. 50A	34F	SM						9.9[e]					23.9	1.0
No. 10A	35	SC						11.9					15.3	9.9
No. 1	36A	SC						14.8					28.0	8.5
No. 42	36B	SC						14.8					28.0	8.5
No. 4	28D	SM						15.9					21.2	11.1
No. 5	28E	SM						15.9[e]					21.2	11.1
No. 41	28F	SM						18.4					23.5	9.5
No. 7	37	SC						23.0					40.0	14.0
No. 55	38	SC							0.24		0.26	0.20		
No. 56	39	SC							0.33		0.41	0.26		
No. 60B	40A	SC							0.48		0.86	0.20		
No. 50B	40B	SC							0.48[e]		0.86	0.20		
No. 61A	41	SC							0.59		0.87	0.28		
No. 63	42	SM								0.39	0.60	0.22		
No. 87	43[c]	SM								0.41	0.70	0.06		
No. 61B	44[c]	SM								0.49	0.70	0.32		
No. 65B	45[c]	SM								0.56	0.72	0.47		
No. 91	46	?								0.60[f]	...	...		
No. 83	47	SM								0.60	0.88	0.25		
No. 60A	48	SM								0.62	1.02	0.06		
No. 57	49	SM								0.72	1.29	0.12		
No. 62A	50	SM								0.76	1.35	0.47		

TABLE I (*concluded*)

Building Unit	Brick Number	Molding Process[a]	Rate of Absorption, g. absorbed in 1 min. or ml. absorbed in 2 min.											
			Low			Moderate			High					
			Chipped Face Average Under 0.03 ml.	Unchipped Face Average Under 0.03 ml.	Average Under 5 g.	Chipped Face Average 0.03 to 0.20 ml.	Unchipped Face Average 0.03 to 0.31 ml.	Average, 5 to 25 g.	Chipped Face Average Over 0.20 ml.	Unchipped Face Average Over 0.31 ml.	Maximum, ml.	Minimum, ml.	Maximum, g.	Minimum, g.
No. 84	51	SM								0.82	1.37	0.43		
No. 85	52	SM								1.05	1.42	0.76		
No. 62B	53[c]	SM								1.30	1.76	0.94		
No. 88	54[c]	SM								1.42	1.80	0.87		
No. 53B	55[c]	SM								1.42[g]	...	...		
No. 90	56[b]	DP								2.09	2.87	1.32		

[a] Molding process: SC—side cut; SM—soft mud; DP—dry press.

[b] Bricks Nos. 5 and 56 occurred together. The top story was brick No. 5 with window trim of No. 56, and the first story was exclusively No. 56.

[c] Common brick.

[d] Average of seven samples. All shipments not tested.

[e] Not tested. Assumed absorption value from tests on other shipments of same brick.

[f] Not tested because brick had been stuccoed. Absorption value estimated from report of ten per cent absorption in one hour immersion.

[g] Not tested because of moistureproofing treatment. Absorption value estimated.

Mortars Studied:

Eight mortar mixes and one masonry cement had been used in the building units studied. The mixes listed in Tables II and III are given in proportions by volume of the cement, lime and sand; or the cement and sand with lime added. The lime added was a percentage of the volume of the cement. All hydrated lime was added dry except in one case. According to the manufacturer's information, masonry cement A in the beginning was a mix by volume of 1.3 parts cement, 1.5 parts normally hydrated lime and 7 parts sand, but was changed later to a mix of 1 part cement, 2 parts lime and 7 parts sand. The latter mix was probably used at building units Nos. 42, 47, 48, and 49. Various standard brands of portland cement were used, and a number of different sands.

Classification of Brick:

Table I shows the classification of the bricks by rate of absorption and gives their molding process. Where the rate of absorption is expressed in grams, the absorption was obtained by placing the dried bricks flat side down in $\frac{1}{8}$ in. of water for 1 min. and measuring the increase in weight. Many of the bricks were tested in place at the buildings by cementing the mouth of a small tubular vessel to the face of the brick and measuring the amount of water absorbed from this vessel in 2 min. If the brick had a fire flashed or rough textured surface, this surface was chipped away and the test made on the surface exposed. The field test used has been described in a previous paper (1), and many of the measurements given in that paper have been used here.

Field results indicated that bricks with a rate of absorption of from 5 to 25 g. in 1 min. could be laid dry with single tooled concave joints, and would produce moistureproof walls if the other factors were favorable. This range of the rate of absorption has been considered moderate; below 5 g. has been considered low; and above 25 g. has been considered high.

TABLE II.—MOISTUREPROOF BUILDING UNITS.

Building Unit	Building Age, yr.	Mortar				Face Brick			Wall Construction			Parapet Wall Treatment	Spandrel Beam Waterproofing	Joint Tooling	Building Height Stories	Thickness Top Story Walls, in.	Extent Of Leaks	Ave. Separation Cracking, per cent
		Mortar Mix	Water Retentivity	Type of Lime[a]	Kind of Lime[b]	Brick Number	Rate of Absorption	Molding Process	Type[d]	Back-Up Material	Walls Furred							
No. 1	10	1:1:4.8[c]	high	Q	HC	36A	moderate	side cut	skeleton or bearing	brick or tile	all	covered	none	concave	2	12 & 16	none	10.6
No. 2	10	1:1:5	high	Q	HC	34A	moderate	soft mud	bearing	brick	all	roofed	none	concave	4	12	none	...
No. 3	8	1:1:5	high	Q	HC	32	moderate	soft mud	bearing	brick	all	covered	none	concave	4	12	none	...
No. 4	7	1:1:5	high	Q	HC	28D	moderate	soft mud	bearing[d]	brick	all	covered	none	concave	2	12	none	13.2
No. 5	6	1:1:5	high	Q	HC	28E	moderate	soft mud	bearing[d]	brick	all	covered	none	concave	2	12	none	16.0
No. 6A	6	1:1:5	high	SH	D	24A	moderate	side cut	skeleton	tile	all	covered	none	concave[e]	2	12	none	...
No. 6B	6	1:1:5	high	SH	D	31B	moderate	side cut	skeleton	tile	all	covered	none	concave[e]	2	12	none	...
No. 7	6	1:1:5	high	SH	D	37	moderate	side cut	skeleton	tile	1 of 3[f]	covered	none	concave[e]	2	12	none	7.0
No. 8	6	1:1:5	high	Q	HC	8	low	side cut	skeleton	tile	2 of 3[f]	covered[g]	none	concave[e]	2	12	none	13.2
No. 9	6	1:1:5	high	SH	D	24B	moderate	side cut	skeleton	tile	all	covered	none	concave	2	12	none	26.0
No. 10A	6	1:1:5	high	SH	D	35	moderate	side cut	skeleton	tile	all	covered	none	concave	3	12	none	9.4
No. 10B	6	1:1:5	high	SH	D	31A	moderate	side cut	skeleton	tile	all	covered	none	concave	3	12	none	9.0
No. 11	7	1:1:5	high	Q	HC	34D	moderate	soft mud	bearing[d]	cinder block	all	roofed	none	concave	1	12	none	10.9
No. 12	6	1:1:5	high	SH	D	34E	moderate	soft mud	cavity	...	none	roofed	none	concave[e]	1	13½	none	4.6
No. 13	9	1:1:5	high	Q	HC	33	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	18.5
No. 14	9	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	9.0
No. 15	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	9.5
No. 16	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	7.3
No. 17	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	21.3
No. 18	9	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	22.1
No. 19	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	7.3
No. 20	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	7.8
No. 21	9	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	13.9
No. 22	8	1:1:5	high	Q	HC	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	6.2
No. 23	6	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	16.8
No. 24	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	19.9
No. 25	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	5.3
No. 26	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	12.3
No. 27	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	39.5
No. 28	6	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave[e]	1	9½	none	11.0
No. 29	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	8.5
No. 30	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	...
No. 31	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	17.3
No. 32	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	23.7
No. 33	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	36.5
No. 34	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	36.5
No. 35	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	...
No. 36	6	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave[e]	1	9½	none	4.0
No. 37	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	21.7
No. 38	7	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	20.3
No. 39	6	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave[e]	1	9½	none	5.2
No. 40	6	1:1:5	high	SH	D	34B	moderate	soft mud	cavity	...	none	roofed	none	concave[e]	1	9½	none	12.7

No. 41......	7	1:1:5	high	SH	D	28F	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	none	13.2
No. 42......	10	Masonry cement A	low	NH	D	36B	moderate	side cut	bearing[d]	cinder block	all	covered[g]	none	concave	1	12	none	22.3
No. 43A....	19	Masonry cement A	low	NH	D	10	moderate	side cut	skeleton	brick	none	flashed	none	concave	2	12	none	4.8
No. 44......	18	Masonry cement A	low	NH	D	22	moderate	soft mud	bearing[d]	tile	all	roofed	none	concave	2	12	none	18.3
No. 45......	17	Masonry cement A	low	NH	D	14A	moderate	side cut	bearing[d]	cinder block	all	covered[g]	none	concave	2	12	none	4.3
No. 46......	17	Masonry cement A	low	NH	D	6	low	soft mud	bearing[d]	cinder block	all	covered[h]	none	concave	2	12	none	28.7
No. 47......	16	Masonry cement A	low	NH	D	20	moderate	side cut	bearing[d]	conc. block	all	roofed	none	concave	2	12	none	...
No. 48......	16	Masonry cement A	low	NH	D	19	moderate	side cut	bearing[d]	conc. block	all	roofed	none	concave	1	12	none	...
No. 49......	16	Masonry cement A	low	NH	D	15	moderate	soft mud	bearing[d]	cinder block	all	covered[h]	none	concave	2	12	none	...
No. 50A....	17	Masonry cement A	low	NH	D	34F	moderate	soft mud	bearing	brick	all	covered[i]	none	concave	4	12	none	...
No. 51......	23	1:3 + 50%	low	NH	?	12	moderate	side cut	bearing[d]	brick	none	roofed	none	concave	2	12	none	5.3
No. 52......	18	1:3 + 10%[j]	low	NH	?	28C	moderate	soft mud	bearing	brick	none	roofed	2nd fl.	concave	2	12	none	14.0

---

[a] Type of lime; Q—quicklime; SH—special hydrate; NH—normal hydrate.
[b] Kind of lime: HC—high calcium; D—dolomitic.
[c] Volumes of dry sand.
[d] Brick and joist construction.
[e] Double tooled; once while soft and again when beginning to stiffen.
[f] One wall with waterproof plaster.
[g] Copper flashing over back and top.
[h] Complete copper cutoff through wall.
[i] Overlapping copper cutoff.
[j] Center of wall poured full of grout each brick course.

TABLE III.—BUILDING UNITS WHICH HAVE SHOWN MOISTURE PENETRATION.

Building Unit	Building Age, yr.	Mortar				Face Brick			Wall Construction			Parapet Wall Treatment	Spandrel Beam Waterproofing	Joint Tooling	Building Height Stories	Thickness Top Story or Leaky Walls, in.	Extent Of Leaks	Average Separation Cracking, per cent
		Mortar Mix	Water Retentivity	Type of Lime[a]	Kind of Lime[b]	Brick Number	Rate of Absorption	Molding Process	Type	Back-Up Material	Walls Furred							
No. 53A....	17	1:1:6	low	NH	?	21	moderate	side cut	skeleton	tile or brick	main[c]	covered	fabric	concave	3	12[d]	small	6.0
No. 53B....	17	1:1:6	low	NH	?	55	high	soft mud[g]	skeleton	brick	none	covered	fabric	flat	3	8	large	...
No. 54......	17	1:1:6	low	NH	?	11	moderate	side cut	skeleton	tile	all	covered	fabric	concave	3	12	small	12.3
No. 55......	17	1:1:6	low	NH	?	38	high	side cut	skeleton	tile	none	covered	fabric	concave	2	12	large	5.1
No. 56......	17	1:1:6	low	NH	?	39	high	side cut	skeleton	tile	all	covered	fabric	concave	1	12	moderate	49.5
No. 57......	16	1:1:6	low	NH	?	49	high	soft mud	skeleton	tile	all	covered	fabric	concave	2	12	very large	26.6
No. 58......	17	1:1:6	low[e]	NPH	D	2	low	side cut	skeleton	tile	all	flashed	fabric	concave	2	12	moderate	18.6
No. 59......	18	Masonry cement A	low	NH	D	14B	moderate	side cut	bearing[f]	tile	all	flashed	none	concave	2	12	small	2.5
No. 43B....	19	Masonry cement A	low	NH	D	26	moderate	soft mud[g]	skeleton	brick	none	flashed	none	flat	2	12	small	19.3
No. 60A....	18	Masonry cement A	low	NH	D	48	high	soft mud	bearing[f]	tile	all	flashed	none	concave	2	12	small	14.0
No. 60B....	18	Masonry cement A	low	NH	D	40A	high	side cut	bearing[f]	tile	all	flashed	none	concave	2	12	small	9.0
No. 50B....	17	Masonry cement A	low	NH	D	40B	high	side cut	bearing	brick	all	flashed	none	concave	4	12	moderate	...
No. 61A....	18	Masonry cement A	low	NH	D	41	high	side cut	bearing[f]	cinder block	all	flashed	none	concave	2	12	moderate	12.8
No. 61B....	18	Masonry cement A	low	NH	D	44	high	soft mud[g]	bearing[f]	cinder block	all	flashed	none	flat	2	12	moderate	42.0
No. 62A....	18	Masonry cement A	low	NH	D	50	high	soft mud	bearing[f]	tile	all	flashed	none	concave	2	12	very large	37.8
No. 62B....	18	Masonry cement A	low	NH	D	53	high	soft mud[g]	bearing[f]	tile	all	flashed	none	flat	2	12	very large	48.5
No. 63......	17	Masonry cement A	low	NH	D	42	high	soft mud	bearing[f]	tile	all	covered[h]	none	concave	2	12	large	51.9
No. 64......	19	1:3 + 50%	low	NH	?	4	low	side cut	skeleton	tile or brick	none	flashed	fabric	concave	2	12	small	22.1
No. 65A....	23	1:3 + 50%	low	NH	?	9	moderate	side cut	bearing	brick	none	flashed	none	flat	5	20	large	...
No. 65B....	23	1:3 + 50%	low	NH	?	45	high	soft mud[g]	bearing	brick	none	flashed	none	flat	5	16 to 20	very large	...
No. 66......	7	1:3 + 33%	low	NH	?	34C	moderate	soft mud	cavity	...	none	roofed	none	concave	1	9½	very large	26.7
No. 67......	20	1:3 + 25%	low	NH	HC	3	low	side cut	skeleton	tile	none	flashed	fabric	concave	20	12	moderate	68.3
No. 68......	23	1:3 + 25%	low	NH	?	7B	low	soft mud	curtain or skeleton	brick or tile	none	flashed	none	concave	2	12 & 16	very large	65.2
No. 69......	23	1:3 + 25%	low	NH	?	7A	low	soft mud	skeleton or curtain	brick or tile	none	flashed	none	concave	2	12 & 16	very large	33.4
No. 70......	23	1:3 + 25%	low	NH	?	7D	low	soft mud	curtain	brick	none	flashed	none	concave	2	12 to 20	very large	52.1
No. 71......	23	1:3 + 25%	low	NH	?	7C	low	soft mud	skeleton or curtain	brick or tile	none	flashed	none	concave	2	12 & 16	very large	...
No. 72......	21	1:3 + 25%	low	NH	?	16	moderate	side cut	skeleton	tile	none	flashed	none	concave	3	12	small	26.2
No. 73......	22	1:3 + 25%[i]	low	NH	D	13	moderate	side cut	skeleton	tile	none	flashed	none	rough cut	3	12	very large	96.0
No. 74......	22	1:3 + 25%[i]	low	NH	?	18	moderate	side cut	skeleton	tile	none	flashed	none	rough cut	3	12	very large	84.5
No. 75......	22	1:3 + 25%	low	NH	?	17	moderate	side cut	bearing	brick	none	flashed	none	concave	5	12 & 16	very large	65.5
No. 76......	21	1:3 + 25%	low	NH	?	23	moderate	soft mud	skeleton	tile	none	flashed	fabric	raked	2	12	large	...
No. 77......	20	1:3 + 25%	low	NH	?	25A	moderate	soft mud	skeleton	tile	none	flashed	fabric	concave	2	12	very large	27.9
No. 78......	21	1:3 + 25%	low	NH	?	25B	moderate	soft mud	skeleton	tile	none	flashed	fabric	concave	4	12	very large	...
No. 79......	21	1:3 + 25%	low	NH	?	28A	moderate	soft mud	bearing or skeleton	brick or tile	none	roofed[j]	fabric	concave	2	12	very large	...

No. 80	18	1:3 + 25%	low	NH	D	28B	moderate	soft mud	skeleton	tile or brick	none	flashed	fabric	concave	5	12 & 16	very large	33.2
No. 81	19	1:3 + 25%	low	NH	?	29	moderate	soft mud	skeleton	tile	none	flashed	fabric	concave	2	12	very large	49.5
No. 82	17	1:3 + 25%	low	NH	?	30A	moderate	soft mud	skeleton	tile	none	covered	fabric	raked	2	12	very large	32.7
No. 83	18	1:3 + 25%	low	NH	?	47	high	soft mud	skeleton	tile	all	flashed	fabric	concave	2	12	small	54.8
No. 84	18	1:3 + 25%	low	NH	?	51	high	soft mud	skeleton	tile	none	flashed	fabric	concave	2	12	very large	31.1
No. 85	19	1:3 + 25%	low	NH	?	52	high	soft mud	skeleton	tile	none	roofed[j]	fabric	concave	2	12	large	48.5
No. 86	20	1:3 + 15%	low	NH	?	30B	moderate	soft mud	skeleton	tile	none	flashed	fabric	raked	2	12	very large	26.0
No. 87	21	1:3 + 15%	low	NH	?	43	high	soft mud[g]	bearing	brick	2 of 3	flashed	none	flat	2	12 & 16	moderate	...
No. 88	19	1:3 + 15%	low	NH	?	54	high	soft mud[g]	bearing	brick	none	flashed	fabric	flat	2	12 & 16	moderate	35.5
No. 89	18	1:3 + 10%[k]	low	NH	?	1	low	side cut	skeleton or bearing	cinder block	none	flashed	fabric	concave	2	12	very large	...
No. 90	18	1:3 + 10%[k]	low	NH	?	5 & 56	low[l]	side cut	skeleton	tile	none	covered[h]	fabric	flat	2	12	small	...
No. 91	20	1:2½ + 10%	low	NH	?	46	high	?	bearing or curtain	cinder block	none	flashed	none	?[m]	2	12	very large	...

[a] Type of lime: NH—normal hydrate; NPH—normal plastering hydrate.
[b] Kind of lime: D—dolomitic; HC—high calcium.
[c] Penthouse walls not furred.
[d] Only penthouse walls show leakage.
[e] Normally hydrated finishing lime soaked 10 days. Water retentivity probably moderately good but not high.
[f] Brick and joist construction.
[g] Common brick.
[h] Copper flashing over back and top.
[i] Grit added. Very unworkable mortar.
[j] Partially roofed, rest flashed. Both sections leaked.
[k] Center of wall poured full with grout each brick course.
[l] Top story built of brick No. 5 with trim of No. 56. First story has brick No. 56. Building considered as if built with brick No. 5 which has low suction.
[m] Entire building was stuccoed to stop leaks.

Figures 1 and 2 show the relation between the amounts of water absorbed in the face tests and the conventional tests in $\frac{1}{8}$ in. of water. The correlation is not all that could be desired. Absorption at the face varies somewhat in each brick, and it was found that the amount of water absorbed in the conventional test might vary as much as 50 per cent or more between the two flat sides of the brick, especially if the bricks were soft burned and torn when the mold was scraped. It appears that the moderate rate of absorption measurement of from

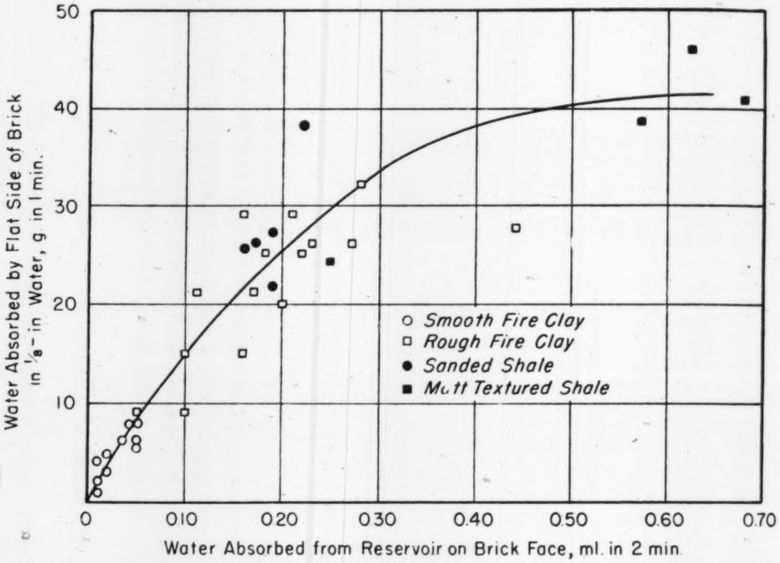


FIG. 1.—Relationship of Tests of Rate of Absorption of Brick by Reservoir on Chipped Brick Face and in $\frac{1}{8}$ in. of Water.

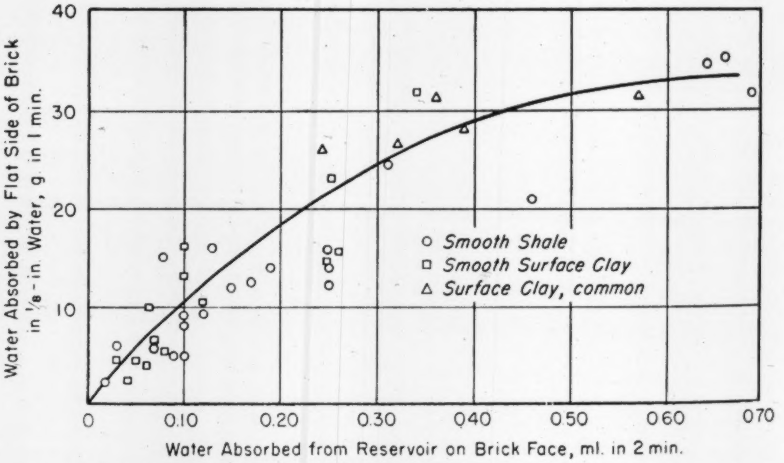


FIG. 2.—Relationship of Tests of Rate of Absorption of Brick by Reservoir on Unchipped Brick Face and in $\frac{1}{8}$ in. of Water.

5 to 25 g. absorbed in 1 min. is a range which corresponds to the range of from 0.03 to 0.20 ml. absorbed by the chipped faces of bricks, and from 0.03 to 0.31 ml. absorbed by the unchipped faces of bricks.

DATA

Tables II and III are all-inclusive and cover the complete study. Table II gives the materials and practices used at the moistureproof building units. Table III gives those used at the building units which showed moisture penetration. This division was based on the records of moistureproofing work. Of the 46 building units that have shown moisture penetration, 45 have been moistureproofed. If it had been found necessary, at any time to point the joints in an area of brickwork at a building unit, the unit has been classed as having shown moisture penetration. There were no records of when these units first leaked, but 38 were treated to stop leaks within six years after construction. Many leaked for some years previous to moistureproofing. Most of the units classed as moistureproof have not leaked since construction, but they have been so classed if they were made moistureproof without the pointing of masonry. Calking was installed four years ago around the wood trim at the entrance and around the frames of small louvered openings at the 31 units with cavity walls, because leaks had occurred at these points in 17 cases. Unit No. 66 leaked a few months later and had to be pointed. The remainder of these units have not leaked since the calking and have been classed as moistureproof.

TABLE IV.—FACTORS WHICH OCCURRED TOGETHER IN THE FIFTY-FOUR MOISTUREPROOF BUILDING UNITS.

Number of Units	Lime Content of Mortar	Parapet Treatment	Mortar Joint Tooling	Rate of Absorption of Brick	Units With All Walls Furred	Units with Water Retentivity of Mortar	
						High	Low
1	high	flashed	concave	moderate	none	none	1
1	low	roofed	concave	moderate	none	none	1
2	high	covered	concave	low	1	1	1
50	high	roofed or covered	concave	moderate	18[a]	42	8

[a] Thirty of the units had cavity walls which were not furred.

DISCUSSION OF DATA

It should be emphasized that the data in this study were obtained from existing buildings. They were not part of a research program designed to establish the facts regarding any of the points in question, nor did they occur by chance. Conclusive proof concerning the influence of any factor, therefore, requires more than the statistical evidence of occurrence.

The causes of a damp spot on a wall can seldom be identified with certainty, but such a spot indicates an opening or a crack in the exposed surface, or a surface material with a porosity which permits water to bleed through. Cracks appear to be the most common cause, but it is generally recognized that there are several factors which cause cracks to occur or otherwise affect the penetrability of a wall. One unfavorable factor may cause leaks, but to produce a moistureproof wall, several of the factors must be favorable to moisture resistance.

Table IV shows how the various factors which occurred at more than 75 per cent of the moistureproof building units, grouped together. Furred walls have been included because 79 per cent of the units with integral walls had furring on all walls.

Moderate rate of absorption bricks, high lime content mortar, a roof or parapet covering, and concave mortar joint tooling occurred together in fifty cases. On the basis of occurrence alone, it appears that these factors were highly favorable to the moisture resistance of the walls and were required in combination to consistently produce moistureproof building units. High water retentivity was a property of the mortar used at 43 of the moistureproof units and apparently was a desirable factor. All walls were furred at 19 of the 24 integral walls units, and this appeared to be a desirable factor. The evidence of occurrence indicates a considerable degree of probability, but conclusive proof requires support. Further evidence is presented under the discussion of each factor.

TABLE V.—DISTRIBUTION OF PARAPET TREATMENTS AND SPANDREL BEAM WATERPROOFING.

	Number of Units	Parapet Wall Treatment		Spandrel Beam Waterproofing	
		Units Roofed or Covered	Units Flashed	Units With	Units Without
Moistureproof units	54	53	1	1	53
Units which have shown moisture penetration	46	12[a]	36[a]	23	23
Totals	100	65	37	24	76

[a] Two units had pitched roofs over part and flashed parapets around remainder of roof area.

Influence of Parapet Treatment:

Units have been classed as roofed only in case a pitched roof covered or eliminated parapet walls. Parapet walls have been classed as covered where the top and back of the parapets were completely covered with metal or felt, or the metal formed a complete cutoff through the parapet. Parapet walls have been classed as flashed where the roof flashings entered the wall for only a few inches, and where the cutoffs or the coverings on the top did not turn down at the outside face to provide complete protection from water entrance. Pitched roofs and covered papapet walls have been considered together because they both protect the wall below from water penetrating at the top.

The data in Table V indicate that protection of the walls by a roof or covered parapets was a factor highly favorable to moisture resistance. Obviously a wall is likely to leak if it is not watertight at the top, and copings are seldom watertight.

Only one of the 37 units with flashed parapet walls was moistureproof, indicating that this was a factor detrimental to the resistance of a wall to moisture penetration. In confirmation, unit No. 59 had all of the apparently favorable factors except that the parapet wall was flashed, the metal flashing not quite covering all of the top of the wall. The top story leaked in several places and small areas of the brickwork had to be pointed. It was apparent that the major source of water penetration was the open tops of the parapet walls.

Influence of Spandrel Beam Waterproofing:

Spandrel beam waterproofing consisted of asphalt and asphalt-impregnated fabric covering for the spandrel beams and was installed as a cutoff through the walls at the roof and floor spandrel beam levels. The outer edge of the membrane, however, always stopped 1 in. or more back from the face of the wall, thus preventing it from being a complete cutoff. In some cases, the membrane stopped back of stone cornices. As shown in Table V, only one unit with spandrel beam waterproofing was moistureproof. The membrane at this unit, No. 52, was not installed at the roof level.

The evidence is strong that spandrel beam waterproofing was a detrimental factor. Units Nos. 53A and 54 apparently leaked because of the spandrel beam waterproofing, although all the other factors were favorable. Cracks formed completely around these units at their roof spandrel beam levels where the membrane turned out to the face of the wall; and the parapet wall movement on the cleavage plane of the membrane caused diagonal cracks at the corners as wide as $\frac{1}{2}$ in. Cracks have occurred at the roof spandrel beam level at all those units with spandrel beam waterproofing which turned out to the face of the wall at that level. It seems significant that there was no visible evidence of parapet wall movement at any of the buildings in this study where there were no cutoffs of metal or fabric to furnish cleavage planes, although one such wall was 206 ft. long, and several were over 100 ft. Building unit No. 73 had no spandrel beam waterproofing and showed no parapet wall movement, but a smaller extension to the rear of similar construction, unit No. 55, with spandrel beam waterproofing, showed excessive parapet movement.

Influence of the Lime Content in the Mortar:

All but one of the 54 moistureproof building units had high lime mortars, indicating that a high lime content probably was a favorable factor. The high lime mortars at these units, however, were in combination with uniformly favorable factors, except in three cases. Twenty units with high lime mortars leaked where other factors were unfavorable. Only one unit with a low lime mortar was moistureproof, but unfavorable factors were always present. Unit No. 66, which leaked, had a low lime mortar with all other factors favorable, but the mortar joints were so poorly filled, this evidence had to be disregarded. The occurrence record alone is not sufficient to establish the influence of the lime content in mortar. A study of the mortar joint cracking supplies additional evidence.

In a previous study (1) it was found that the high lime mortars consistently produced less separation cracking than the high cement (low lime) mortars. A comparison of the mortar joint cracking at units in the present study, having moderate rate of absorption bricks, integral walls, and concave tooled joints, shows that the cracking at 16 units averaged 11.1 per cent with high lime mortars, and averaged 36.1 per cent at six units with low lime mortars. The maximum cracking of 26.0 per cent at unit No. 9 with a high lime mortar, was bettered only by one low lime mortar at unit No. 52, which had a cracking of 14.0 per cent. The difference in cracking with these two types of mortars is significant.

It is generally agreed that mortar joint cracking is a cause of moisture penetration, and one of the basic principles of moistureproofing is to eliminate the cracks at the mortar joints by pointing or other means, if they are numerous. Forty-five of the 46 leaky building units in this study have been moistureproofed. Persistent wall leaks were finally stopped at six of the units by additional pointing of the mortar joints in areas where leaks had recurred in spite of partial pointing, the covering of parapets where unprotected, calking where necessary, and the application of transparent waterproofing. The stopping of these recurrent leaks solely by additional pointing of the joints is good evidence that the joint cracking was their cause

Unit No. 52 had a low lime, low water retentivity mortar, but the center of each brick course was poured full with grout. The brick suction was moderate; the mortar joint cracking was much less than at any other unit with a low lime mortar, and there were no leaks. This may indicate that this mortar, combined with the grouting procedure, would produce moistureproof walls with moderate suction bricks, if the other factors were uniformly favorable. Units Nos. 89 and 90 had a similar mortar with low suction bricks. Both leaked.

The low percentage of joint cracking produced by high lime mortars and the excellent record of these mortars in the moisture-proof units indicate that the high lime mortars used were a highly favorable factor in the moisture resistance of the walls.

The high percentage of separation cracking produced by the low lime mortars used and their high incidence in the leaky units indicate that these mortars were a detrimental factor in moisture resistance.

The Influence of Water Retentivity in Mortar:

The ratings of high or low water retentivity given the various mortars studied were, to a degree, assumed, because no tests were made of the mortars before use. However, various tests have indicated that normally hydrated lime used dry seldom produces high water retentivity mortar. Neither the manufacturer of the normally hydrated dolomitic lime most frequently used in the units, nor the manufacturer of masonry cement A, claimed that their product would produce high water retentivity mortar. On the other hand, the two high plasticity quicklimes and the two brands of specially hydrated limes used have generally produced mortars, of the proportions used in the units, which tested high in water retentivity. The influence of the various sands used is uncertain but there were indications that differences in sand may explain some of the wide variations of the mortar joint cracking in similar combinations of brick and mortar.

Table VI shows that all the high water retentivity mortars were in moistureproof units, but eleven of the moistureproof units had mortars with low water retentivity. The mortar joint cracking at units with integral walls, moderate rate of absorption bricks, concave tooled joints, and high lime mortar, averaged 12.5 per cent at eight units having high water retentivity mortars, and 9.5 per cent at eight units having low water retentivity mortars. The average cracking increased to 36.1 per cent for low retentivity mortars at six units, similar except that the mortars had a low lime content, indicating the influence of the lime content. It seems significant that high lime content was a factor common to all the mortars except one in the moistureproof units.

TABLE VI.—DISTRIBUTION OF THE LIME CONTENT AND THE WATER RETENTIVITY OF THE MORTARS.

	Number of Units	Units With High Lime Content Mortars	Units With Low Lime Content Mortars	Units With High Water Retentivity Mortars	Units With Low Water Retentivity Mortars
Moistureproof units	54	53	1	43	11
Units which have shown moisture penetration	46	20	26	0	46
Totals	100	73	27	43	57

High water retentivity in mortar was probably a desirable factor because it eliminated very low plasticity limes, but there was no good evidence at the units studied that low water retentivity was an unfavorable factor if the lime content of the mortar was high. It is believed, however, that none of the limes used in the high lime mortars had a very low plasticity.

Influence of the Rate of Absorption of Brick:

As shown in Table VII all but two of the moistureproof building units had bricks with a moderate rate of absorption, indicating that this was a favorable factor, but all of the accompanying factors were also favorable, except in one case. There were bricks of high, low, and moderate suctions at the leaky units, but each unit had some other factor that was unfavorable except unit No. 63. This unit had high rate of absorption bricks but all other factors were favorable. It leaked badly and had a mortar joint cracking of 51.9 per cent, indicating that the high rate of absorption of the brick was a detrimental factor. A comparison of the mortar joint cracking at units with integral walls, high lime mortars, and concave tooled mortar joints showed that the average cracking at 16 units was 11.1 per cent for the moderate suction bricks, 20.7 per cent for the low suction bricks at four units, and 25.8 per cent for the high suction bricks at eight units. The general practice was to wet down all high suction bricks with a hose except in freezing weather, and this should have reduced the suction at the time of laying in most cases.

The evidence is strong that a moderate rate of absorption for brick was a highly favorable factor in moisture resistance. It is also indicated that a high rate of absorption was a detrimental factor.

The evidence regarding the influence of low rate of absorption bricks is not entirely conclusive. Various laboratory investigators (**5**, **8**, **9**, **10**) have found that low rate of absorption bricks produced the least permeability in panels, and when the joints were filled, this kind of brick generally gave the best results with high cement (low lime) mortar. Palmer (**5**) expressed doubts about the low rate of absorption bricks giving the same results in walls of buildings where the joints might not always be completely filled with mortar, and pointed out that this type of brick had been widely associated with leaky buildings. The field data in this study show that the seven units with low suction bricks and low lime mortars leaked, and the extent of leaks at five was very large. All of these units had one or more other factors which have been indicated as detrimental, and these were probable causes of leaks, but the average mortar joint cracking at four units was 54.8 per cent which is very high. Of the four units with low rate of absorption bricks and high lime mortar, two were moistureproof, and two leaked. The two leaky units had two other factors that were detrimental, but the joint cracking averaged 20.4 per cent which is high. The extent of leaks was small or moderate. At unit No. 8 the first part of a wall built with the 3.8-g. brick and the high lime, high water retentivity mortar showed such a high percentage of joint cracking that the practice of double tooling the joints was adopted and the cracking was materially reduced. The

TABLE VII.—DISTRIBUTION OF BRICKS BY RATE OF ABSORPTION AND MOLDING PROCESS.

		Rate of Absorption of Bricks			Molding Process	
	Number of Units	Moderate Units	Low Units	High Units	Side Cut Units	Soft Mud Units
Moistureproof units	54	52	2	0	14	40
Units which have shown moisture penetration	46	18	9	19	18	27[a]
Totals	100	70	11	19	32	67

[a] One unknown.

average cracking of the double tooled joints was 13.2 per cent. Nine units leaked out of the total of eleven units with low rate of absorption bricks, but all of the leaky units had other factors that were detrimental to moisture resistance.

The evidence is somewhat inconclusive, but the high percentage of units with low rate of absorption bricks which leaked, and the high percentage of mortar joint cracking which occured with these bricks and both low lime and high lime mortars, indicate that low rate of absorption bricks were a detrimental factor. It is also indicated that if all other factors are favorable and the surface skin of the bricks has some absorption, moistureproof walls can be built with low suction bricks. Five grams absorbed in one minute, however, appears to be the minimum safe limit for rate of absorption, but double tooling may reduce this.

The Influence of Mortar Joint Tooling:

All of the moistureproof units and 31 of the leaky units had concave tooled mortar joints. The mortar joints at nine of the leaky units were flat tooled, two were rough cut, and three were raked. The indications are that concave tooling was a highly favorable factor, but it cannot make walls moistureproof if other factors are unfavorable. Conditions favorable to a reasonable comparison of the mortar joint cracking with flat and concave tooling were present at units Nos. 43A and 43B. Both these units had moderate rate of absorption bricks and the same high lime mortar and wall construction. The cracking at No. 43A with concave tooling was 4.8 per cent, and at No. 43B with flat tooling was 19.3 per cent. Unit No. 43B leaked. Rough cut mortar joints showed abnormally high cracking, and the average cracking at two units with raked joints was 29.4 per cent, which is high.

The evidence of the data is not conclusive, but concave tooling appeared to be a highly favorable factor in moisture resistance. All of the other types of tooling studied are judged to have been detrimental.

At nine moistureproof units, the joints were concave tooled first while quite soft and then tooled a second time when the mortar started to stiffen. The average cracking at seven of these units was 8.2 per cent, which compared with an average of 15.9 per cent at 37 moistureproof units with single tooled concave joints. Double tooling undoubtedly is a desirable practice, especially if the bricks have a suction slightly outside the desirable range of 5 to 25 g. absorbed in 1 min.

Influence of Wall Furring:

The wall furring was of two types, metal lath and channels and wood furring strips with wall board, both of which were a base for plaster. The construction industry has very positive opinions concerning wall furring, but the data indicate that furring had a limited value. Thirty of the moistureproof units had cavity walls which were not furred. Nineteen of the 24 moistureproof integral wall units had all walls furred, but five were unfurred or had only part of the walls furred. Fourteen of the 45 leaky units with integral walls had all walls furred; 29 had no furring and 2 units were partially furred.

Obviously, furring did not always prevent moisture penetration when unfavorable factors were present, but there are indications at six units that the extent of leaks was reduced by furring. However, the extent of leaks was large or very large at four of the furred units. It is also evident that furring was not always necessary to produce moistureproof walls. Furthermore, of the 29 unfurred units that leaked, 22 have been made moistureproof and the leaks at 3 others reduced to minor areas by remedial treatments applied to the outside face of the wall only. One was furred to stop leaks, but the leaks recurred in several areas.

It appears that the influence of wall furring was to reduce the extent of leaks at units where detrimental factors were present, but it seldom prevented leaks from occurring. It apparently was a desirable practice.

Influence of Type of Wall:

Thirty of the 31 units with cavity walls were moistureproof, which was much the best record of any type of wall, but all the moistureproof cavity wall units had all of the highly favorable factors and none of the detrimental factors. Nine of the moistureproof units had skeleton walls and 16 had bearing walls, and two each of the skeleton wall and bearing wall units had an unfavorable factor. The indications were that any of these types of wall could be built moistureproof if the other factors were favorable. None of the five units with curtain walls were moistureproof but all had three unfavorable factors. The leaky units give no satisfactory evidence that any particular type of wall was an unfavorable factor. The data furnish no convincing indication that the type of wall exerted a significant influence on moisture resistance.

Influence of Wall Thickness:

There were cavity walls in this study with thicknesses of 9½ in. and 13½ in., all of which were moistureproof except one. There were 24 moistureproof units with 12-in. integral walls; and there were walls up to 20 in. thick in the units that leaked. A wall thickness of 20 in. was not sufficient to make a unit moistureproof if detrimental factors were present. It is indicated, however, that an integral wall thickness of 12 in. was sufficient for moistureproof construction if the other factors were uniformly favorable.

Influence of Back-Up Materials:

In the moistureproof units with integral walls, brick back-up material was used at nine units, tile at nine, cinder block at five, and concrete block at two. The units that leaked had all of these back-up materials except concrete block. There is no evidence to indicate that the back-up material exerted any important influence on the resistance to moisture penetration.

Influence of Building Height:

There were 21 moistureproof units of from two to four stories in height, and 33 were one story structures. In general, the exposure of a building tends to increase with its height, but eleven of the one-story units were located at the seashore where the exposure was probably more severe than at taller buildings farther inland. Apparently the leaks that occurred at the units studied were caused by factors other than the height. The evidence covered only four stories, but there was nothing to indicate that moistureproof walls could not be built to much greater heights if all the other factors were favorable.

Influence of the Type of Lime in the Mortar:

All of the building units which leaked were built with mortars containing normally hydrated lime, but this does not appear significant because eleven of the moistureproof units had mortars with a similar lime. There were also 17 quicklimes and 26 specially hydrated limes used in the moistureproof units. Moistureproof units were obtained with all types of lime where the other factors were favorable to moisture resistance. The type of lime apparently exerted no significant influence.

Influence of the Kind of Lime in the Mortar:

The records were not complete for the kind of lime used in the mortars. Two were unknown in the moistureproof units, and 32 were unknown in the leaky units. There were 35 known dolomitic limes and 17 high-calcium limes used in moistureproof units. Moistureproof walls were produced with mortars containing either kind of lime if the other factors were favorable. The data do not indicate any important influence.

Influence of the Molding Process of Bricks:

As shown in Table VII, 14 side-cut bricks and 40 soft-mud bricks were used in the moistureproof units, and 18 side-cut bricks and 27 soft-mud bricks were used in the units that leaked. The molding process of one was unknown. It appears that the molding process of the brick exerted no significant influence.

Indications of the Separation Cracking Between Brick and Mortar:

The measurements of the separation cracking at 32 of the units were made in a previous study (1), and the method of making such measurements was there described. Measurements at 46 other units have been added. The cracking was not measured at all of the units, but enough measurements are available to furnish good evidence. The average cracking at 44 moistureproof units was 14.7 per cent (14.7 ft. of cracks per 100 ft. of mortar joints), which compares with an average cracking of 36.3 per cent at 34 units which have leaked, or with an average of 32.7 per cent for 26 leaky units with concave tooled joints similar to the moistureproof units. The joint cracking at the moistureproof units was less than half of that at the units which have leaked.

The average separation cracking has been used in several cases as an indication of the relative merits of different materials and practices. The number of units compared has sometimes been small, but the differences in average cracking were so great that the possibility of misleading indications is remote.

The Use of Calking Compound:

No data on the use of calking compound have been included in this study because the materials used and the joints calked varied so greatly at the different units that such data would have been more misleading than not. The stone trim, window frames, and joints between masonry and metal were calked at all of the specially designed building units to eliminate the cracks that generally form at these points, but such complete calking practices were not followed at the remainder of the moistureproof units nor at the leaky units. It appears, however, that lack of calking was seldom, if ever, the sole cause for moisture penetration at the leaky units, because all but six of these units leaked after remedial calking or in areas of brickwork between a calked coping or a covered parapet and stone trim on the wall. Calking is highly desirable at certain joints to prevent cracks in the wall surface, but the details of design determine its necessity.

Report on Buildings Specially Designed to Prevent Moisture Penetration:

Forty-three buildings, units Nos. 1 to 42 inclusive and unit No. 66, were designed with special emphasis on moistureproof construction. The efficacy of the measures used have been tested by exposure to the weather for from 6 to 10 yr. Several have been subjected to the extremes of seashore exposure.

The materials and practices selected for these buildings were the result of a 10-yr. study of the problem of leaky masonry walls by the author of this paper. The major sources of leaks had been determined from field studies and observations, and remedial practices had been developed in moistureproofing work. The basic principles of moistureproof construction, and the materials and practices that were factors in moisture resistance had been tentatively established from a synthesis of the indications of field experience and studies of existing buildings, combined with the findings of laboratory investigators. The practices and materials selected were those which promised the best combination for moistureproof walls. They were incorporated into the specially designed new buildings, first as a test, but later because of the good results obtained.

All of the buildings have been moistureproof since construction with only one exception, unit No. 66. At this building the contractor failed to fill the mortar joints and did not use the high-lime mortar specified. It leaked badly and the walls had to be pointed extensively. At 16 of the small cavity wall buildings, minor leaks developed around the wood trim at the front entrances or at the frames of small openings, but these were stopped by calking. None has leaked in the four years since calking, showing that the brick walls were moistureproof.

The units had many variables. They varied in height, in the type and thickness of walls, the back-up material used, the type and kind of lime in the mortar, and the molding process of the brick, but only one had any factor indicated in this study to be detrimental to moisture resistance.

All of the 42 moistureproof buildings in this group had the main walls protected by roofs or covered parapets, all but one had moderate rate of absorption bricks, and all had high-lime mortars, concave tooled mortar joints and mortars with high water retentivity. With two exceptions, the buildings with integral walls had all walls furred. The fact that these significantly favorable factors of practices and materials produced 42 successive buildings with all walls moistureproof in spite of many other variables is convincing proof that, in combination, they prevented moisture penetration.

The Combinations of Factors Needed to Produce Moistureproof Walls:

Table IV shows how the factors favorable to moisture resistance occurred in combination in the moistureproof building units. It required at least three of the highly favorable factors to produce any of the moistureproof building units in this study. Four of the moistureproof units had combinations of three of the highly favorable factors, but six of the units that showed leaks also had three of these factors in combination.

To produce moistureproof brick walls consistently, it was apparently necessary to have a combination of all four highly favorable factors of high lime mortar, protection of the top of the walls by a roof or covered parapet walls, moderate rate of absorption bricks, and concave tooled mortar joints. Fifty of the units with this combination of factors were moistureproof, and only two leaked.

Influence of Detrimental Factors:

The effect of detrimental factors was apparent. The leaks at the two units, Nos. 53A and 54, which had all of the four highly favorable factors in combination, were apparently caused by spandrel beam waterproofing. Unit No. 63 with three highly favorable factors and furred walls apparently leaked because of high rate of absorption bricks. Unit No. 59 with three highly favorable factors and furred walls apparently leaked due to flashed parapet walls. Only four units with the indicated detrimental factors were moistureproof. All of the units that leaked had one or more of the apparently detrimental factors of spandrel beam waterproofing, flashed parapet walls, high or low rate of absorption bricks, low lime mortar, and flat tooled, raked, or rough-cut mortar joints. Detrimental factors occurred 132 times, and in 127 of these instances they were associated with units that leaked.

SUMMARY AND CONCLUSIONS

The data in this paper were obtained from 100 existing building units, subject during construction to the variables of individual workmanship, weather conditions, and all the other variables normal to field work. The units were exposed to the severe storms of a seacoast state for periods ranging from 6 to 23 yr. Many widely used materials and practices were not included in the study. The conclusions apply only to the building units studied and the materials and practices used, and only to brick masonry walls above grade. In particular, the combination of factors reported here as the minimum for moistureproof wall construction should *not* be regarded as the only possible combination.

The following conclusions appear to be justified:

1. It was found that the protection of the top of walls by a roof or by covering the top and back of parapet walls or by complete parapet cutoffs; the use of the mortars studied with a lime content equal to or greater than 50 per cent of the volume of the cement; the use of bricks with a rate of absorption between 5 and 25 g. of water when set flat side down in $\frac{1}{8}$ in. of water for 1 min.; and the concave tooling of mortar joints, were all factors highly favorable to the moisture resistance of brick masonry walls.

2. The use of mortars with high water retentivity and the use of wall furring were desirable factors in obtaining resistance of brick masonry walls to moisture penetration.

3. The separation cracking between brick and mortar was reduced by concave tooling the mortar joints while soft, and retooling them when the mortar had begun to stiffen.

4. The use of spandrel beam waterproofing; the use of flashings which did not cut off or completely cover the top and back of the parapet walls; the use of bricks having a rate of absorption greater than 25 g. when set flat side down in $\frac{1}{8}$ in. of water for 1 min.; and, except where grout was poured into each brick course, the use of mortars with a lime content less than 50 per cent of the volume of the cement, were factors detrimental to the resistance of brick masonry walls to moisture penetration.

5. The use of bricks having a rate of absorption less than 5 g. of water when set flat side down in $\frac{1}{8}$ in. of water for one minute apparently was a factor detrimental to the moisture resistance of brick masonry walls, but the evidence was not conclusive.

6. Rough cut, raked, or flat tooled mortar joints were judged to be detrimental to the moisture resistance of brick walls, but the evidence was sparse.

7. There was no satisfactory evidence that any significant influence on the moisture resistance of brick walls was exerted by the kind and type of lime, the molding process of the brick, the kind of material used to back up integral walls, the height of the walls up to and including four stories, or the thickness and type of the walls. The minimum thickness of moistureproof integral walls, however, was 12 in.

8. The average separation cracking between the mortar joints and the bricks in the moistureproof building units was less than half of that at the units which showed moisture penetration.

9. The importance of the use of calking compound as an aid to moistureproof wall construction was stated.

10. A report on the results obtained

at a group of 43 buildings designed with special emphasis on moistureproof construction shows that all but one were moistureproof, and that the specifications were not followed in the construction of the one building which leaked.

11. The data show that the minimum combination of favorable factors, which consistently produced moistureproof walls, consisted of the four factors listed in Conclusion 1.

12. The detrimental factors listed in Conclusions 4, 5, and 6 were associated with units that leaked with but few exceptions.

Acknowledgments:

The author wishes to acknowledge his indebtedness to the skilled craftsmen and engineers in the fields of moistureproofing and brick construction who contributed so greatly to the solution of the problem of leaky walls. Especially is he indebted to L. A. Palmer, formerly Research Fellow at the National Bureau of Standards, and Walter C. Voss of Massachusetts Institute of Technology, for their pioneer research on the properties of mortars and bricks and the nature of bond. Their findings greatly influenced the selection of the brick and mortar in 1937 for the walls of the group of specially designed buildings, the moistureproofness of which have indicated the primary importance of the materials and practices used.

REFERENCES

(1) C. C. Connor, "Resultant Separation Cracking Between Various Mortars and Brick in Existing Brick Structures," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 454 (1934).

(2) L. A. Palmer, "Water Penetration Through Brick-Mortar Assemblages," *Journal*, Clay Products Inst., Vol. I, p. 19 (1931).

(3) L. A. Palmer and D. A. Parsons, "The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. XXXV, No. 18, p. 18 (1932).

(4) L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Journal of Research*, National Bureau of Standards, Research Paper No. 683, Vol. 12, May, 1934.

(5) L. A. Palmer and D. A. Parsons, "Permeability Tests of 8-In. Brick Wallettes," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 419 (1934).

(6) W. C. Voss, "Permeability of Brick Masonry Walls—An Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

(7) F. O. Anderegg, "Construction of Watertight Brick Masonry," *Journal*, Am. Ceramic Soc., Vol. 13, p. 351 (1930).
F. O. Anderegg, "Watertight Brick Masonry," *Archt. Record*, Vol. 70, p. 201 (1931).

(8) C. C. Fishburn, D. Watstein, and D. E. Parsons, "Water Permeability of Masonry Walls," BMS7, U. S. Govt. Printing Office, October 18, 1938.

(9) C. C. Fishburn, "Water Permeability of Walls Built of Masonry Units," BMS82, U. S. Govt. Printing Office, April 15, 1942.

(10) John W. McBurney, M. Arnold Copeland, and Russell C. Brink, "Permeability of Brick-Mortar Assemblages," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1333 (1946).

DISCUSSION

MR. WALTER C. VOSS[1] (*presented in written form*).—It is extremely reassuring to consider the findings of Mr. Connor in this paper, fifteen years after the writer's first attempt to define the requirements for the production of watertight brick masonry. In June, 1933, the writer proposed an hypothesis in a paper presented before the Society[2] as follows:

> "A leakless brick masonry wall may be produced by an assemblage of brick and mortar where, first, the relation of the absorptive power of the brick and the amount of water in the mortar is such that the initial movement toward the brick plane sets in early and continues at an even rate for several hours; and where, second, the mortar is so constituted as to permit such loss of water and still provide a soluble product which in the process is concentrated at the brick plane and is available thereafter for a long period of time to form a compound with a minimum of differential shrinkage with the brick, the assemblage being capable by its porous structure to permit repetition of such action."

Since that time many studies have been made by various investigators, but few of these have been as thoroughly and practically done as those represented by Mr. Connor's paper.

A few of the underlying factors will be reviewed to recall the important rôle lime plays in this problem. The first of these is the prompt response of the lime hydrates and carbonates to the action of water. The accompanying Fig. 3 shows the rate of expansion of several

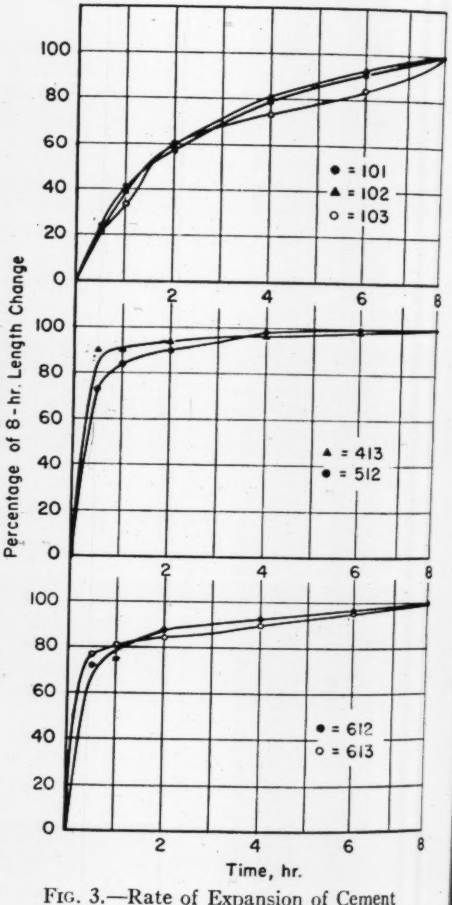


FIG. 3.—Rate of Expansion of Cement and Cement-Lime Mortars.

101, 102, 103—1 cement—0 lime—2½ sand
413—1 cement—1 lime—5 sand
512—1 cement—1½ lime—6¼ sand
612, 613—1 cement—2 lime—7½ sand

---

[1] Head, Department of Building Engineering and Construction, Massachusetts Institute of Technology, Cambridge, Mass.

[2] W. C. Voss, "Permeability of Brick Masonry Walls—An Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

cement and lime-cement mortars. It will be noted that the rigid siliceous cement mortar lags and that the higher lime mortars promptly expand. This aids in preventing the entrance of excesses of water.

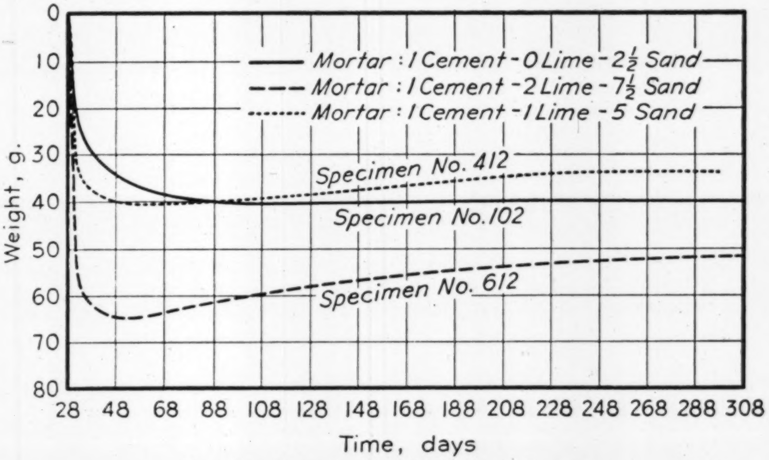


FIG. 4.—Comparative Weight Loss Curves.

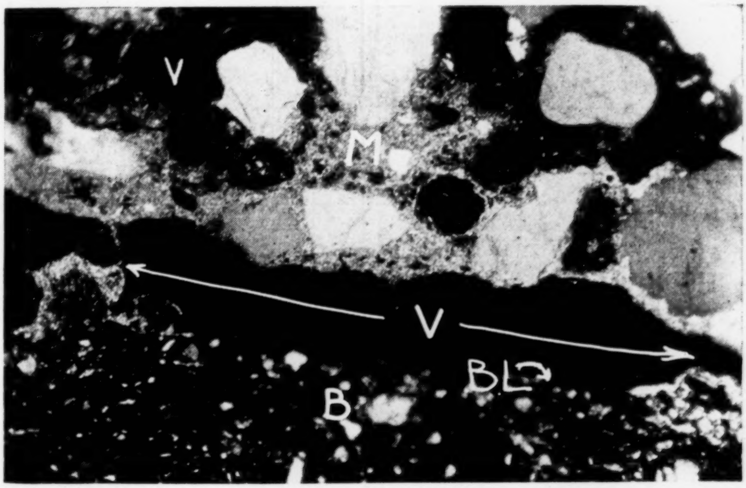
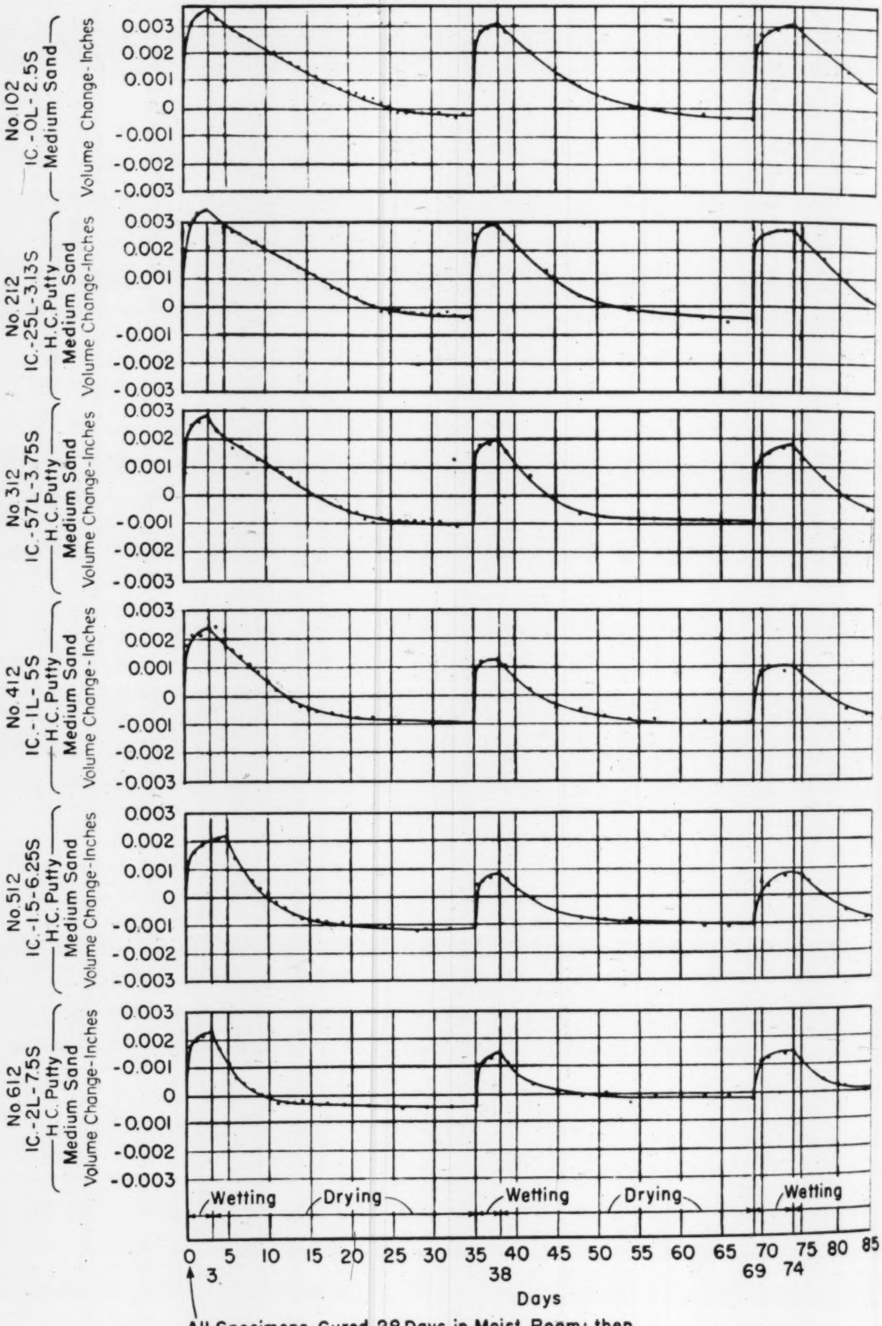


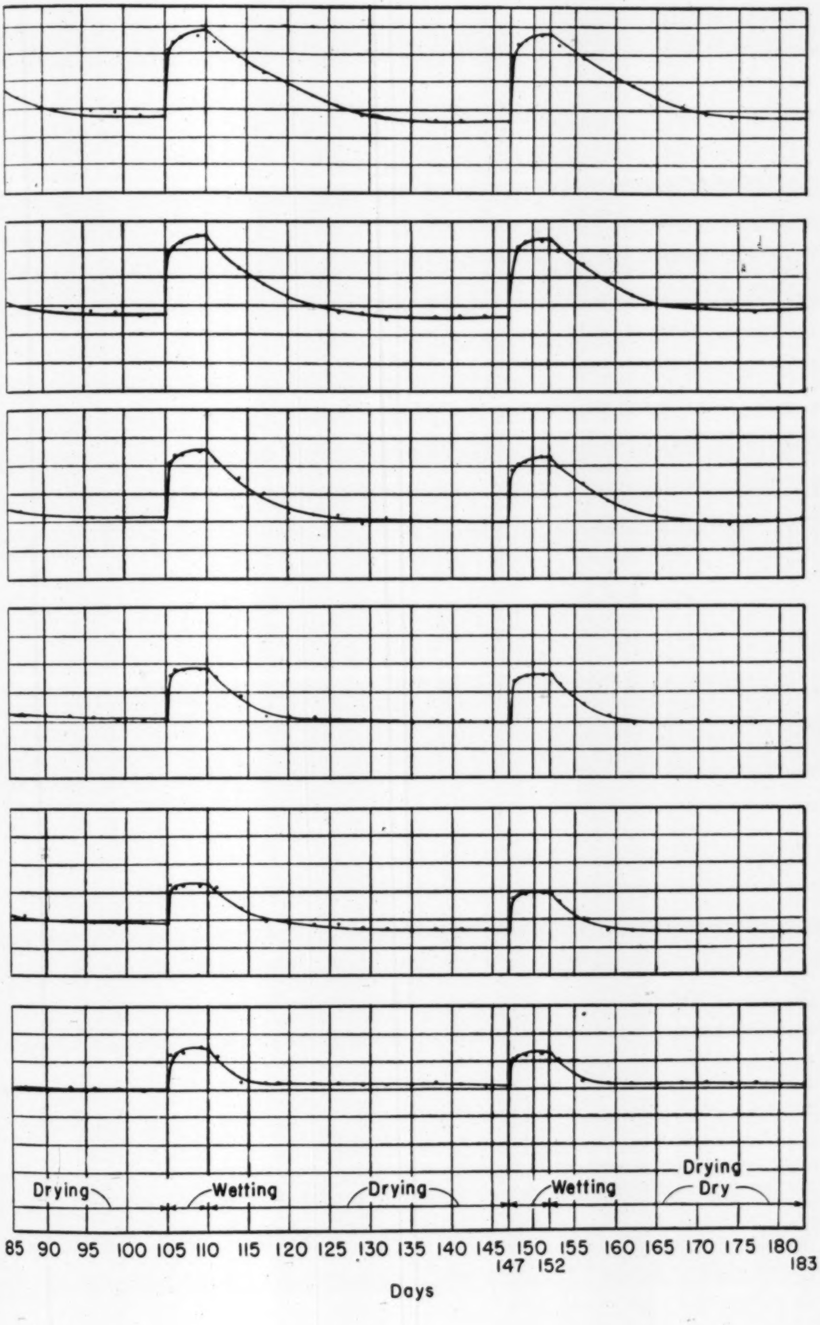
FIG. 5.—Interface Condition for a Cement Mortar and Relatively Dense Brick.

The second of these is the gradual stabilization and reduction in volume change which occurs with the high-lime mortars after successive cycles of wetting. Figure 6 shows the wetting and drying effects on various mortars over 5 cycles, covering 183 days after stabilization for 298 days at 80 F. and 50 per cent relative humidity. It will be noted that the cement mortar follows a more or less unvarying pattern much the same in magnitude from cycle to cycle, while the high-lime mortars gradually decrease in the magnitude of change and



All Specimens Cured 28 Days in Moist Room; then Stabilized in 80F.- 50 Per Cent Relative Humidity Room Until 298 Days Old.

FIG. 6.—Wetting and Drying



Effects on Various Mortars.

react very quickly. This again aids in reducing differential effects which are so important in preventing separation cracking.

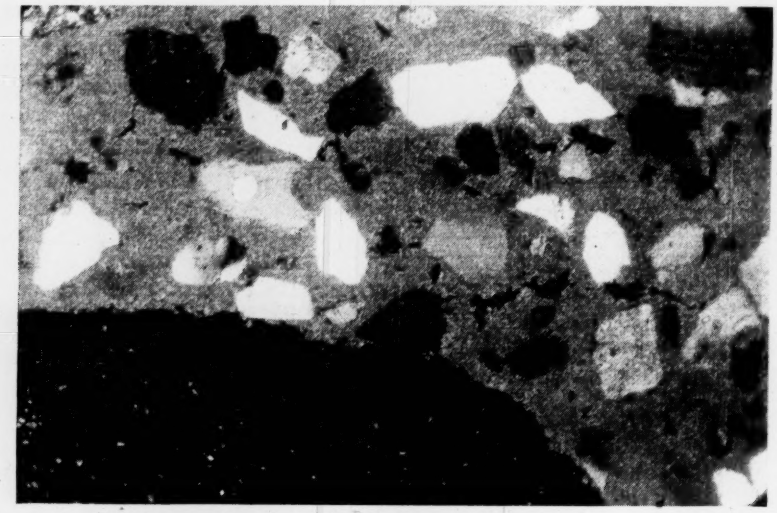


FIG. 7.—Contact Between Lime Mortar and a Highly Absorptive Brick.

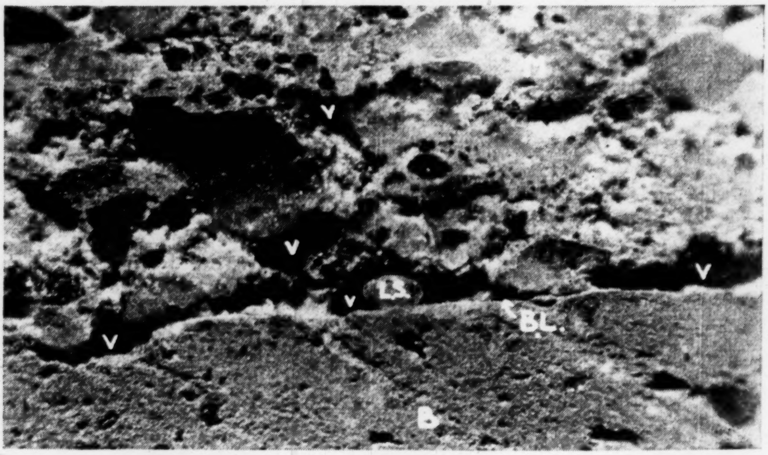


FIG. 8.—Section of Cement Mortar and Moderately Absorptive Brick.

The third is the increase in weight and consequent density which occurs in high-lime mortars after successive wettings. Figure 4 shows the effects of alternate wetting and drying in cycles of approximately 35 days each, over a period of 183 days. It will be noted that the change in weight of the cement mortar is practically zero while the lime-cement mortars increase in weight as the proportion of lime is increased. This means that CO_2 has transformed $Ca(OH)_2$ into $CaCO_3$ which gradually seals many pores. Cement mortars are not effective in this regard and thus are partially responsible for separation crack-

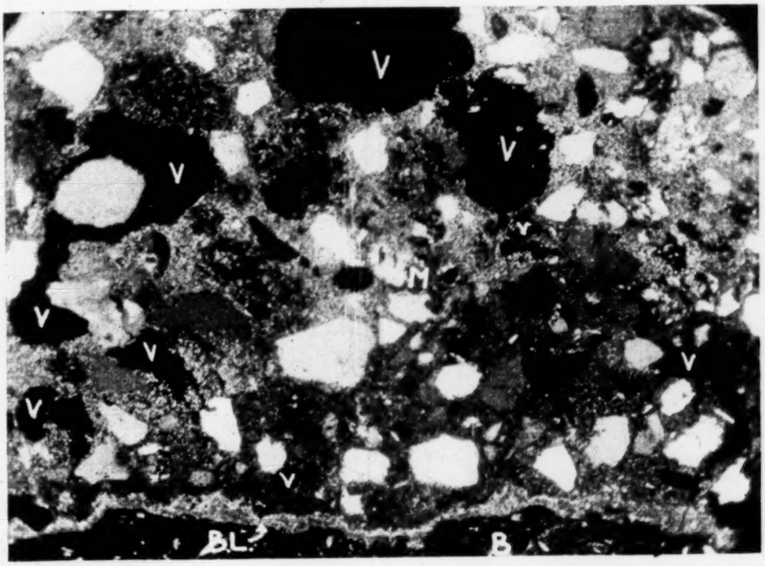


FIG. 9.—Specimen, 109 years old, Composed of Lime Mortar and Moderately Dense Brick Polarized Light.

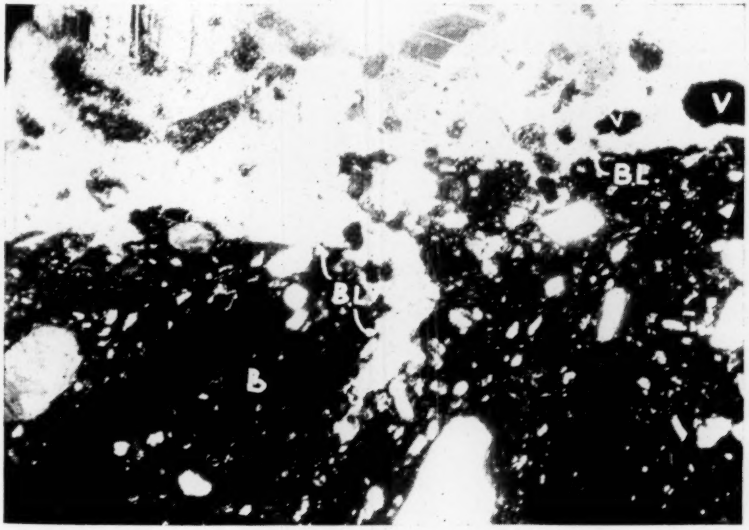


FIG. 10.—Lime-Cement Mortar.

ing which is almost directly proportional to their loss of water.

As to the appearance of the interfacial planes when cement mortars and lime-cement mortars are used, reference is made to five of the photomicrographs selected from many which illustrate similar tendencies.

In Fig. 5 are shown the interface conditions for a cement mortar and a relatively dense brick, photographed by reflected light at 20 diameters. This specimen was four years old at the time the photomicrograph was made. The typical, tentacular contact exhibited by cement mortars is shown, which is often due to low absorptive brick and bleeding of the mortar. This condition prevents continuity of contact and aggravates leakage at the interface.

In Fig. 7 is shown the intimate contact between a lime mortar and a highly absorptive brick. This specimen was 187 years old when the section was made and was photographed under polarized light at 60 diameters.

Figure 8 shows a combination photographed by reflected light at 20 diameters from a section of cement mortar and moderately absorptive brick which was 9 years old. This again indicates the lack of intimate contact. In this case, the condition was probably due to excessive extraction of water from the mortar by the brick.

Figure 9, a specimen 109 years old, which was composed of a lime mortar and a moderately dense brick, was photographed under polarized light at 60 diameters. This specimen shows that separation cracking was present at first, but dissolution and redeposition has built a layer of carbonate into the interstices. The wall did not leak at the time the sample was taken.

Figure 10, a specimen four years old, was photographed under polarized light at 60 diameters. This was a lime-cement mortar and illustrates the ease with which the plastic mortar entered the irregularities of the brick surface, and some of this material was quite probably built into the voids by dissolution and redeposition.

In conclusion, it seems reasonable to say with Mr. Connor that more than 50 per cent of lime by volume of cement is one of the necessary requirements for tight brick masonry walls.

MR. C. C. FISHBURN[3] (*presented in written form*).—The term "separation cracking" in Mr. Connor's paper implies the formation of cracks between the brick and mortar after the completion of the wall. Cracks may result from severe frost action, unequal settlement of the foundations and restraints induced by the foundation or adjacent structural members. Such cracks are readily discernible at some distance from a wall, may greatly increase the permeability of a wall, but are not further included in this discussion.

Other cracks in brick masonry may possibly result from changes in the temperature and moisture content of the masonry which tend to produce differential volume changes between the brick and the mortar. However, tests[4] indicate that such volume changes and the resultant cracking, if any, have no important effect on the permeability of brick masonry test walls.

Tables II and III of Mr. Connor's paper are of value because they contain data on the construction of water permeability of existing buildings. Excepting cavity wall constructions the average amount of "separation cracking" listed in Table II for 16 buildings that did not leak was about 13 per cent. For Table III the average amount of cracking in eight buildings that showed small leakage was 18 per cent, the average cracking in nine buildings that showed moderate to large leakage was 37 per cent and the average cracking in fifteen buildings that showed very large leakage was 47 per cent. Obviously there was a definite relationship between the amount of "separation cracking" noted by Mr.

---

[3] Materials Engineer, National Bureau of Standards, Washington, D. C.

[4] Building Materials and Structures Reports, U. S. Government Printing Office:
Report BMS 41, "Effect of Heating and Cooling on the Permeability of Masonry Walls,"
Report BMS 55, "Effects of Wetting and Drying on the Permeability of Masonry Walls,"
Report BMS 76, "Effect of Outdoor Exposure on the Water Permeability of Masonry Walls."

Connor and the amount of leakage in the buildings. A possible reason for this relationship is presented below.

Brick masons usually adjust the consistency of the mortar to the absorptive properties of the bricks; they prefer to use a "stiff" mortar for bricks that do not rapidly absorb water. When high-absorptive units are laid without previous wetting, the suction of the brick will quickly stiffen the mortar. Mortar also tends to stiffen on the board before it is used, the amount of drying and stiffening depending on the elapsed time and on the weather. Now the use of a too stiff mortar may have two results. It will increase the permeability of the walls by reducing the adhesion or bond between the brick and the mortar, and it will reduce the workability of the mortar thereby affecting the continuity of the junction between the faces of the brick and the faces of the mortar joints. For rough cut joints this lack of continuity is well known and the tooling of joints containing a stiff mortar may not entirely correct this condition.

Possible reasons for the correlation between the cracking and permeability data in Tables II and III of Mr. Connor's paper are listed as follows:

1. Most of the "separation cracking" observed by Mr. Connor was not a true cracking, did not occur after the walls were built and the defects were built into the masonry at the time the brick were laid.

2. A mortar that was too "stiff" was used in many of the buildings listed as having leaked, and the use of a "stiff" mortar was an important factor affecting the permeability of the buildings.

3. The discontinuity or apparent cracking between the edges of the brick and the mortar joints was increased by the use of mortars that had dried and stiffened.

It would be interesting to know if Mr. Connor has any records or data on this matter.

MR. J. W. MCBURNEY[5] (*presented in written form*).—Accepting the conclusions of Fishburn and his coworkers[6] that "workmanship affected the permeability of the walls more than any other factor," the writer of this discussion had not expected that Mr. Connor's data would so well confirm the results of the several laboratory[6, 7, 8] studies in which workmanship was controlled. That all walls laid with mortar of high water retentivity (lime putty or special hydrate) were moisture proof and all walls that were not moisture proof were laid with mortar of low water retentivity (normal hydrate) seems quite in accord with Fishburn's[6] finding that "the use of a lime of low plasticity producing a mortar with low water retentivity greatly increased the permeability of the walls—." Were there any water retentivity determinations on the mortars made from masonry cement A of Table II? More information on masonry cement A may supply the "satisfactory evidence" for judging the influence of kind and type of lime on moisture resistance. (Mr. Connor's conclusion No. 7.) The data of Watstein[9] and Fink and Trattner[10] well uphold Mr. Connor's assumption that cement-lime mortars made with special hydrate have high water retentivities. There are good and poor putties made from quicklime, and the probabilities are somewhat against the ordinary mason's hydrates rating about 65 per cent. However, unless their quality is as poor as the "lime C" reported by Wells and his coworkers[11] a 1:2:7 mortar might meet the 65 per cent requirement. It is unfortunate that Mr. Connor's data did not include results on high water retentivity 1:$\frac{1}{4}$:3 mortars, more 1:2:7 mixes, and some masonry cements with water retentivities exceeding 80 per cent and made without lime. Mr. Connor's definition of a high-lime mortar as one with more lime than 1:$\frac{1}{2}$ cement lime apparently has created some confusion. Most of us confine the term to mortars richer in lime than 1:1 by volume, as, for example, a 1:2:9 or 1:3:12.

---

[5] In Charge, Research on Masonry Materials, National Bureau of Standards, Washington, D. C.

[6] C. C. Fishburn, D. Watstein, and D. E. Parsons, "Water Permeability of Masonry Walls," Building Materials and Structures Reports BMS 7, U. S. Government Printing Office, October 18, 1938

[7] C. C. Fishburn, "Water Permeability of Walls Built of Masonry Units," Building Materials and Structures Report BMS 82, U. S. Government Printing Office, April 15, 1942.

[8] J. W. McBurney, M. A. Copeland, and R. C. Brink, "Permeability of Brick-Mortar Assemblages," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1333 (1946).

[9] D. Watstein and N. A. Seese, Jr., "Properties of Masonry Mortars of Several Compositions," ASTM BULLETIN, No. 147, August, 1947, p. 77.

[10] G. J. Fink and E. Trattner, "Properties of Highly Hydrated Dolomitic Masonry Limes and Certain of Their Cement-Lime Motars," *Proceedings*, Am. Soc. Testing Mat., Vol. 45, p. 723 (1945).

Mr. Connor reports as highly favorable the use of bricks with initial rates of absorption between 5 and 25 g. Mr. Fishburn (conclusion 7 of BMS 82) mentions the range 0 to 20 g. for his "excellent" and "good" results. He, however, gives ratings of "fair" and "poor" for some walls built with bricks in the 13 to 20 g. range. Other walls in this same range rated "good." The data quoted are for 8-in. walls built with "A" workmanship and laid with mortar of from 70 to 85 per cent water retentivity. The rating "very poor" applied to walls built of "B" workmanship irrespective of brick suction. The writer's[8] permeability tests did not include any bricks with average penetrabilities between 8 and 26 g., but some unpublished data on bond strength indicate a 50 per cent reduction in strength in shear as penetrabilities of bricks increase from 10 to 20 g. Very few samples of bricks appear to fall into the 10 to 20-g. range. It has been necessary to measure and sort large shipments of bricks averaging above 20 g. or below 10 g. to obtain a few specimens within this range, according to the writer's experience in testing brick from some 300 plants. Considering the variability of bricks and mortar and laboratories, and workmanship, agreement between laboratory and field conclusions that differs by only 5 g. seems to this observer very gratifying.

Mr. Connor's observations on cracking are very interesting. In the absence of photographs or preferably a joint inspection tour, we may not be discussing the same phenomena. It is a matter of observation that cutting mortar joints tends to pull mortar out from between the bricks. Unset mortar has some tensile strength (cohesion) and some ductility, and "necking" occurs as the mortar is pulled away by the trowel. As shown by dye tests, tooling a previously cut joint only partially seals these openings, and breaking and cracking of the feathered edges of the tooled surface is one observed source of cracking. In agreement with Mr. Connor, the amount and width of these "edge effects" vary with different bricks and different mortars. A number of different properties of mortar and bricks may influence the occurrence and magnitude of these openings. Unknown and hitherto unmeasured properties may enter. A laboratory investigation should supply the answer.

It appears obvious that at least half of Mr. Connor's walls were built using better workmanship than Mr. Fishburn's[7] "B" method, It is hoped that the author's closure will include a description of workmanship or workmanships in the terms of those described in BMS 82. The results on cavity walls (30 nonleaking, 1 leaking) confirm the generally held belief that this type of construction is almost workmanship proof.

---

[11] L. S. Wells, D. L. Bishop and D. Watstein, "Differences in Lime as Reflected in Certain Properties of Masonry Mortars," *Journal of Research*, Nat. Bureau Standards, Vol. 17, December, 1936, pp. 895–907 (*RP*952).

In conclusion, the writer considers that Mr. Connor has rendered a very useful service in presenting his data. His paper is unique and most valuable.

MR. H. C. PLUMMER.[12]—The reports from England, British research stations and others seem to indicate cavity wall construction is a major factor in preventing rain penetration. I note that Mr. Connor's data seem to verify that, but in his conclusions he does not list type of construction as an important factor. Why does he not do that?

MR. VOSS.—I should like to say that you can use high absorption brick or low absorption brick that are not entirely impermeable and make a tight wall.

MR. PLUMMER.—I should like to indorse that statement.

MR. F. O. ANDEREGG.[13]—Mr. Connor has made a valuable contribution to the building industry in summarizing his observations on masonry walls. A point or two, however, warrant a little discussion. It is suggested that the words "lack of contact" would better express what Mr. Connor is talking about than "separation cracking." Mr. Connor has never offered any positive proof that such cracking is progressive, as has been intimated. Experimental evidence should be obtained by properly controlled tests; he should erect panels or piers and in sufficient number to get data of statistical value and paint the joints with water soluble dyes at different ages. Then the joints should be broken apart and the areas covered by the dyes should be actually measured.

What is the meaning of the sentence on page 13, "Unit No. 52 had a low-lime, low water retentivity mortar, but the center of each brick course was poured full of grout?"

MR. J. M. HARDESTY.[14] (*by letter*).—Mr. Connor has, in presenting this information on his field observations on 91 brick masonry buildings, given to the profession the kind of data which are needed so much in order to correlate laboratory findings and the application of these principles in actual construction. The value of the paper will be enhanced if it stimulates other engineers to observe and report on the masonry buildings which come under their observations. It is only after a relatively large amount of such field data has been collected that a really true picture can be obtained regarding the effects of the many factors which enter into the construction of moistureproof buildings.

A study of Tables II and III brings out very forceably certain facts which have not heretofore been properly emphasized in constructing brick buildings to resist moisture penetration. These are: (*a*) the use of bricks which do not have too high or too low a rate of absorption; (*b*) the use of a mortar which does not lose its plasticity too easily, that is, mortar with high water retentivity properties; and (*c*) means for preventing water from entering the walls back of the moisture resisting wall facing. There are differences of opinion regarding the limits to be placed on the physical properties of the brick and mortar in (*a*) and (*b*). There are also differences of opinion as to whether these properties of the brick or the mortar are the more important. Regardless of what the upper and lower limits of these properties should be, it appears that some progress might be made in constructing moistureproof buildings if these materials were selected having properties somewhere near the ranges recommended in this paper.

The problems involved in (*c*), above,

---

[12] Director, Engineering and Research, Structural Clay Products Institute, Washington, D. C.

[13] Director, Building Materials Research, John B. Pierce Foundation, Raritan, N. J.

[14] Bell Telephone Laboratories, Inc., Murray Hill, N. J.

are usually structural in character, rather than a choice of building materials. The places where water can enter the walls are numerous and occur not only in the parapet, but at any place where there is a break in the wall. Mr. Connor's criticism of spandrel beam waterproofing may not be entirely justified when the building as a whole is considered. His observations which led to classifying spandrel beam waterproofing as an unfavorable factor were almost all limited to the top stories where roof slab and parapet wall movements cause stresses in the masonry which are not present in other parts of the building. It is probably true that the cleavage in the masonry caused by the waterproofing membrane allows cracks to form more readily at the roof line. However, the data presented do not prove that spandrel beam waterproofing at lower points in the building will fail to deflect to the exterior water which may enter the wall back of the moistureproof facing.

Concave tooling, which to a limited extent pushes the struck mortar back into the joint, is good practice and helps to prevent the formation of ledges to catch water as may happen in flat tooled and raked joints. Just how effective this operation is and just how much it contributes to the making of a moistureproof wall is questionable. From studies[15] which the writer made on factors which affect the strength and extent of bond between brick and mortar, it was found that regardless of the physical characteristics of the mortar and brick and the type of tooling no bond or an intermittent bond occurred frequently at the edge of the brick. This lack of bond occurred at the bottom as well as at the top of the mortar joint and varied in depth up to a third of the width of the brick. Similar brick-mortar interface separations were observed by McBurney, Copeland, and Brink.[8] Although the joints in the moistureproof buildings appeared to be tight, there may be an area of separation back of the tooling which may develop into an open crack after the mortar has weathered for a few years. This observed area of no adhesion of the mortar to the brick tends to throw doubt on the effectiveness of double tooling and the judging of the favorable factors for moistureproof walls by measuring the extent of separation cracks.

Mr. C. C. Connor (*author's closure by letter*).—It is evident from the discussions that the synonymous terms "separation cracking" and "mortar joint cracking," are not well understood. These terms as used cover all the visible cracks and separations between brick and mortar, except the long running cracks obviously due to movement in the structure. Separation cracks are short and discontinuous, and while they may join together horizontally for 3 or 4 ft., they do not extend vertically for more than the height of a single brick. The measurements of these cracks were made in areas free from obvious movement cracks.

Undoubtedly a minor part of the separation between brick and mortar was due to the mortar slumping or being pulled away from the brick in cutting off the joint, but where joints are carefully tooled, almost all of the separation cracking occurs some time after tooling. At units Nos. 7 and 8 the first brickwork laid showed no appreciable separation or cracking shortly after being laid up, but a check the next morning showed at least 20 per cent. Double tooling reduced the cracking considerably in later brickwork.

It appears that separation cracking grows for the first 7 or 8 yr. of a building's life if the bond between the brick and mortar is poor, but it does not grow if the bond is good. There is little recorded data on this growth, but cracking has been studied for over 20 yr. through repeated examinations of many buildings to determine the remedies needed for leaks or to check on how the bond was standing up in various brick and mortar combinations. At unit No. 81, with moderate suction bricks, a low-lime mortar and excellent workmanship, the cracking at six months was estimated at 5 per cent; it grew to about 20 per cent at two years, and was 50 per cent at four years. The area of leakage also expanded. Leaks generally grow in extent and severity for several years. Most moistureproofing treatments on new buildings have had to be extended later to cover larger areas or other walls. This apparently has been due to the growth of separation cracking. The growth of cracking between spot pointing on horizontal joints has been frequently observed. However, at unit No. 1 where the bond was good and no leaks occurred, measurements have been made periodically in the same areas for the ten years of that building's life, and the cracking has remained at 10 per cent, plus or minus 1 per cent. But if excessive water enters a wall, it appears that even good bonds can be destroyed and separation cracks developed.

---

[15] Unpublished data on the strength and extent of bond between brick and mortar made at Bell Telephone Laboratories in 1941.

The amount of separation cracking is a practical indicator used by all expert waterproofers in estimating the amount of pointing necessary and the thoroughness of the treatment needed to stop the evident leaks. There is no question that to prevent moisture penetration the brick and mortar combination should be such as to reduce the separation cracking to a low percentage.

Mr. Hardesty has expressed doubts about the amount of separation cracking being an indication of the influence of a mortar or a brick on moisture resistance because he found in his experiments that a high percentage of cracks occurred with all brick and mortar combinations tested. I strongly suspect this cracking was due to a poorly graded sand, because I have observed unusual cracking at several buildings where the sand was not well graded although the brick and mortar combinations were good. According to the sieve analysis kindly furnished me by Mr. Hardesty, the sand he used had 27 per cent passing the No. 50 sieve, and this was 47 per cent of the sum of the fractions passing the No. 16 but retained on the No. 50 sieve. That is a very high ratio of the fine to the medium-sized particles and normally produces what the masons call a "dead" sand. On the basis of limited data, it appears that a practical measure of a good mortar sand is that it have a fraction passing the No. 50 sieve which is not less than 18 per cent and not greater than 33 per cent of the sum of the medium fractions between the Nos. 16 and 50 sieves. Furthermore, the sum of the fractions retained on the No. 16 sieve should not exceed 25 per cent of the total. The sieve analyses of several sands which have been consistently rated as excellent by master masons, and which have produced low separation cracking at several buildings, show that the fine, medium, and coarse particles were within the limits given above, and that the sand retained on the No. 4 sieve did not exceed 1 per cent. The units in the study with poorly graded sand are not accurately known, but the number where the cracking measurements were affected probably did not exceed ten. However, the influence on separation cracking of bricks and mortars having different properties was clearly indicated in this study and in my previous study on that subject[16] and Mr. Fishburn has pointed out in his discussion how the amount of cracking in my study tended to correlate with the extent of leaks at the building units. The evidence seems clear that the separation cracking does indicate the influence of a brick or mortar on moisture resistance. Nevertheless, it appears that Mr. Hardesty's discussion has served to develop the importance of the influence of sand grading. The probability of such influence was commented upon in the paper, but we need to know more about it. I think it is worth noting that Mr. Hardesty's measurements of flow after suction have been consistently low in comparison with other laboratories. Again I suspect the sand used exerted an important influence.

---

[16] C. C. Connor, "Resultant Separation Cracking Between Various Mortars and Brick in Existing Brick Structures," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 454 (1934).

Mr. Fishburn suggests that too stiff mortars may have been the cause for the moisture penetration at the building units. The low-lime mortars studied would normally be called too stiff and the high rate of absorption bricks undoubtedly tended to produce an early stiffening of the low water retentivity mortars. Occasionally, poorly graded sand apparently had the same effect. In these cases the stiffness of the mortar was a product of the properties of the masonry materials. The resulting leaks should be attributed to the materials and not to the stiffness of mortar they produced. I have not noticed the preference of masons for stiff mortar with low suction bricks.

Mr. McBurney is very much mistaken in his assumption that the field data show support for the finding of Fishburn[6] that "the use of a lime of low plasticity producing a mortar with low water retentivity greatly increased the permeability of the walls." To so interpret the field evidence that all of the leaky units had low water retentivity mortars, and all of the high water retentivity mortars were in moistureproof units, is pure sophistry in the light of all the data. The eleven moistureproof units with low water retentivity mortars refute the claim that such mortar increase the permeability of walls. This evidence cannot be so blandly ignored. These units were moistureproof because they had high-lime mortars or had the joints poured full of grout, and because the other factors were almost always favorable to moisture resistance. The controlling thing was whether the other factors were favorable or detrimental. The moistureproof units with high water retentivity mortars had all of the factors favorable except in one case. The leaky units with low water retentivity mortars always had factors which were detrimental. The water retentivity of the mortar exerted no evident influence where the other factors were favorable. The sum of all the evidence on this subject apparently indicates that high water retentivity in mortar is an advantage only where the bricks have a suction outside of the range of from 5 to 25 g., and particularly where they are above that range. And then it only serves to reduce the water penetration unless special measures are taken. Undoubtedly there is a limit for workability below which good results cannot be expected, but water retentivity and workability are not the same thing.

Mr. McBurney's assumption that the field results confirmed the findings of the laboratory where the workmanship was controlled has to be accepted with qualifications, and especially in regard to the studies he mentioned. Laboratory workmanship is generally excellent unless deliberately made otherwise, but different laboratory investigators have reached divergent conclusions. The field results confirmed the conclusions of those who found that a high-lime content in mortar was beneficial, and also confirmed the usual laboratory indications that high rate of absorption bricks increased the moisture penetration. However, the field results showed that low suction bricks and low-lime mortars were detrimental to moisture resistance, which is contrary to the results in the research studies referred to by Mr. McBurney. Also, and to my surprise, the field results minimized the importance of water retentivity in mortar which frequently has been stressed by investigators. The difference in regard to water retentivity was apparently due to the fact that the combination of 5 to 25 g. bricks, with a high-lime, low water retentivity mortar and good workmanship has rarely been tested in the laboratory. When it has been, its good results have been buried in the averages. The other disagreements were probably due to the differences in practice between field and laboratory.

Mr. McBurney points to the fact that 30 of the 31 cavity wall units were moistureproof, and concludes therefrom that this type of wall is almost workmanship proof. There is no excuse for such specious reasoning in view of the other data given. This study has clearly demonstrated that the occurrence record by itself proves very little. The 30 moistureproof cavity wall units had the full combination of the four highly favorable and no detrimental factors, which always produced moistureproof walls in the units studies, regardless of the type of wall. Why, then, credit the moistureproofness of the cavity wall units to their wall type? The one cavity wall unit which leaked had a detrimental mortar factor and poorly filled joints. I have received reliable reports of leaks at over 20 cavity wall buildings in areas not covered by my study. The wall ties and the frames around openings bridge the cavity and water penetrates at these points if the wall face is open. It is time the construction industry stopped spending time and money chasing the mirage of a single cure-all for leaky walls. The water must be stopped in the outside inch or two of the wall. It if gets by that barrier, except by capillarity, moisture penetration is almost certain to occur unless special flashings are used to shed the water out again. The leaking cavity wall unit was made moistureproof by pointing the cracked mortar joints for a depth of $\frac{1}{2}$ in. The unfilled joints are still unfilled but the building does not leak. The same is true for many other moistureproofed buildings.

Good workmanship in finished brickwork means that the joints are filled and mortar faces well tooled. The workmanship at the units studied was variable, but probably was seldom as good as Fishburn's[7] workmanship "A", and never as poor as his workmanship "B". Pointing work and demolition of walls have always shown some unfilled joints or open spaces, except at unit No. 67. This unit leaked.

As explained in the paper, Masonry Cement A was a mixture of portland cement and a widely used, normally hydrated dolomitic lime. Its lime content was high, but the manufacturer admitted its water retentivity was not. It gave excellent results where the other factors were favorable.

Mr. Voss feels that my field data corroborates his estimate of the importance of the bond layer which he discovered.[2] I agree. The concept of a continuous "bond layer" as a requirement for full extent of bond, and produced by liberal amounts of lime in the mortar, has been of great service in selecting mortar materials. It also provides a reasonable answer for the effectiveness of high-lime mortars with moderate suction bricks regardless of their water retentivities.

I agree with Messrs. Voss and Plummer that it is possible to build moistureproof walls with either high or low suction bricks. The results, however, are uncertain, and there is a limit beyond which it is definitely unsafe to go. Good results with high suction bricks apparently require a high-lime, high water retentivity mortar with a well-graded sand, very careful or double tooling, thorough wetting of the brick, and the laying of the bricks with the pick and dip method, or with a spreadout of mortar limited to three bricks. Even then, very hot weather caused considerable joint cracking to develop at a recent building. Freezing weather, which prevented wetting of the brick, produced much worse results at other buildings. Moistureproof buildings have been built with low suction bricks, but the evidence is against them. The chance of getting a good, continuous mortar bond with bricks having highly impervious flat surfaces seems to be poor unless they are laid bone dry and with unusual pressure. These are difficult conditions to meet in field construction. The favorable factors listed in this paper include no unusual procedures, conditions, or workmanship, and tend to make good workmanship easier. If perfection of workmanship, or special requirements have to be depended on to get moistureproof walls, leaks are a probability. As one who is interested primarily in getting moistureproof buildings, I am against any material which reduces the factor of safety. Both high and low suction bricks are a hazard. A varied selection of moderate suction bricks has been obtained without difficulty.

In regard to Mr. Anderegg's question as to what was meant by the statement that the center of each brick course at unit No. 52 was poured full of grout, this practice is not uncommon. The bricks are laid with a full bed, but the other joints are only partially filled with mortar. All unfilled joints in the face brick course and the backup are poured full with cement grout as each full course is completed. Dry cement is then dusted on the top before the next course is laid.

SYMPOSIUM ON METHODS AND PROCEDURES USED IN IDENTIFYING REACTIVE MATERIALS IN CONCRETE

INTRODUCTION

The chemical reaction of aggregates in concrete has been of considerable interest and concern for several years. Numerous instances are known of unfavorable reaction of certain combinations of cement and of aggregate which has been the cause of cracking failures in large concrete structures. Many papers have been written discussing the possibility of reaction between alkali set free in the hydration of cement and certain minerals occurring in the concrete aggregate.

Research has been pointed toward, ways and methods of identifying potential reactive materials before they are actually in combination in a concrete mass. Two technical committees of A.S.T.M.—Committee C-1 on Cement and Committee C-9 on Concrete and Concrete Aggregates—have a vital interest in this field of research from the standpoint of their respective interest in cement and aggregates as well as their common interest in concrete. Subcommittees in both have inaugurated extensive studies and for the past two years have been arranging for joint meetings at which forums were conducted including the presentation of papers on methods of testing reactive materials.

There are a number of ways of identifying potentially reactive materials. However, none of them has been developed sufficiently to warrant consideration as suitable for an A.S.T.M. Standard. Several of these methods have had extensive practical use and now seems a good time to compare them for simplicity of determination and correlation with actual findings in the field to the end that a suitable method may be standardized by the Society. Committee C-9 has arranged the present symposium in the hope that the facts revealed will be of assistance in the preparation of a standard method or methods.

There are two papers written by Bailey Tremper and Thomas E. Stanton, respectively, dealing with the correlation of laboratory tests with field experiences. Both papers describe the use of mortar bar expansion tests and their correlation with field experience. In one case good durability is indicated when low-alkali cement is used with a certain source of aggregates and the other the potential adversely reactive properties of a portland cement—fine aggregate combination.

The paper presented by D. O. Woolf and T. R. Smith describes a simple and rapid method of testing mortar or concrete for the alkali-aggregate reaction by the Mason jar test. This method was developed to offset the principal disadvantage of the mortar bar expansion tests which is that of the time required to complete a test. An acceleration of the reaction by the alkali in the cement and the aggregate was accomplished by the addition of one per cent of sodium hydroxide by weight of the cement to the mortar.

The methods used by the Bureau of Reclamation are referred to in the paper presented by R. C. Mielenz and L. P. Witte. These methods comprise (*a*) petrographic examination and analysis, (*b*) a chemical test with NaOH solution, (*c*) tests of mortar and concrete containing the aggregate under investigation and cements of various alkali content, and (*d*) field and laboratory investigation of concrete in structures.

The petrographic identification of reactive material is described by Byrant Mather. It is suggested in this paper that the techniques of petrography be used to determine the presence or absence of any of the known reactive materials in concrete aggregates as well as providing a check on the results obtained by other tests.

CORRELATION OF LABORATORY TESTS WITH FIELD EXPERIENCES OF EXCESSIVE CONCRETE EXPANSION INDUCED BY A REACTION BETWEEN THE CEMENT AND AGGREGATE*

By Thomas E. Stanton[1]

Synopsis

Seldom is it possible to establish as positive a correlation between laboratory tests and field experience as has been established by the Sealed Container—Mortar Bar Test, in general use for determining the potential adversely reactive properties of a portland cement—fine aggregate combination.

This paper lists a number of typical cases of failure of concrete structures through an adverse reaction between cement and aggregate, where the field experience was in each case subsequently checked by laboratory tests.

In a study conducted to determine the potentially reactive properties of fine aggregates from widely distributed sections of the state, in some cases, where no concrete deterioration had been reported but laboratory tests indicated potential reactivity, subsequent field inspection confirmed the suspected activity in structures in which the same aggregates had been used with a high-alkali cement.

Laboratory Test Procedure

A number of tests have been proposed for the detection of the presence of minerals in an aggregate which may adversely react with the alkali constituents of portland cement. The so-called sealed-container method, substantially as described by the author before the Society[2] is of proven value and, as it has been in use since the first work was done on this phenomenon in 1938–39, as described before the American Society of Civil Engineers[3] it has the most extensive background on which to base a correlation between laboratory tests and field experience.

Because accelerated dependable results are obtained when the specimens are stored in an oven in sealed containers at 100 to 110 F., California procedure has been standardized at 100 F. The use of the term sealed container simply means that as ordinarily conducted, a tight or friction top cover on the container in which the specimens are stored (with a small amount of water in the bottom of the container) is sufficient seal. It is unnecessary to take any other precautions such as soldering or otherwise tight sealing the container between measurement periods.

Mielenz, Green, and Benton have reported in the *Journal* of the American

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Materials and Research Engineer, California Division of Highways, Sacramento, Calif.

[2] Thomas E. Stanton, "Studies to Develop an Accelerated Test Procedure for the Detection of Adversely Reactive Cement-Aggregate Combinations," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 875 (1943).

[3] Thomas E. Stanton, "Expansion of Concrete Through Reaction Between Cement and Aggregate," *Proceedings*, Am. Soc. Civil Engrs., December, 1940, p. 178.

Concrete Institute[4] the details of a chemical test which has considerable merit and may be found on further investigation to be superior to the measurement of the expansion of specimens in the sealed-container test. However, although it may subsequently be determined that sufficient correlation with field experience can be established solely on the basis of a chemical test, this discussion relates entirely to California experience with the change in volume test which has proven and generally accepted merit.

FIELD EXPERIENCES CHECKED BY LABORATORY TESTS

Bradley Pavement Failure:

Early in 1938, a section of concrete pavement north of the town of Bradley in the Salinas Valley, Monterey County, Calif., failed because of excessive expansion.[3] An intensive field and laboratory investigation followed to determine the causes of the failure and to develop safeguards to prevent a recurrence of the condition in future work. The failed pavement was built in the fall, winter, and spring of 1936–37. Most of it had passed through two winters when portions began to show distress in the spring of 1938. The distress became manifest in the form of excessive expansion which caused buckling at the expansion joints and a severe cracking throughout the length of certain slabs.

The fine aggregate came from two sources: one a local deposit (Oro Fino), a dry wash tributary to the Salinas River, and the other (Coyote) a commercially operated deposit near San Jose, Calif. in the San Francisco Bay Region. The sands from these two deposits were used during alternate days so that it was easy to identify the source of the fine aggregate in each section or day's run. An inspection of all sections readily disclosed that excessive expansion had occurred only in those sections in which the local (Oro Fino) fine aggregate had been used, and it was obvious, therefore, that the fine aggregates from this area contained at least one of the sources of trouble.

Earlier Failures:

The Bradley pavement failure culminated a series of concrete failures along the coast area from Monterey County in the north to the northern part of Los Angeles County in the south, throughout which aggregates of the same typical characteristics are encountered.

The first structure in which distress developed to such an extent as to attract attention was the King City Bridge built in 1919–1920 across the Salinas River at King City, Monterey County.

The coarse aggregate was imported from an unqestioned source, but the fine aggregate was taken from the bed of the Salinas River, or a small tributary, Pine Creek, which empties into the river a short distance upstream from the bridge.

Within three years after construction, well-defined cracks were in evidence in the caps of the piers. Later, these cracks extended into the columns of the piers. By July, 1924, all of the pier caps were more or less affected.

The King City Bridge has been emphasized because it was the first partial failure of a major structure to attract attention. Subsequent to the King City Bridge trouble, several concrete trestles in Monterey County, in which fine aggregate from the Salinas River or its tributaries had been used, developed serious distress of a nature similar to that observed in the King City Bridge.

Other pavement and structure failures were noted in Los Angeles, Ventura,

---

[4] Richard C. Mielenz, Kenneth T. Green, and Elton Benton, "Chemical Test for Reactivity of Aggregates with Cement Alkalies; Chemical Processes in Cement-Aggregate Reaction," *Journal*, Am. Concrete Inst., Vol. 19, No. 3, November, 1947; *Proceedings*, Vol. 44, p. 193.

Santa Barbara, San Luis Obispo, and Monterey Counties, where the aggregates were all of the same general type, containing from 4 to 15 per cent of shale, cherty shale, and chert. Several of the more important failures were sections of pavement and sea walls in Ventura and Santa Barbara Counties.

The solution of the problem was complicated by the fact that, although without exception cracking of the nature described was noted only when local fine aggregate had been used, there were numerous instances where the same local aggregate performed in an entirely normal and satisfactory manner.

Immediately north of the failed Bradley pavement is a section of pavement, constructed at least six years earlier, in which fine aggregate from the Salinas River was used, but which is still (after 18 yr.) in excellent condition and shows no evidence of excessive expansion or cracking.

Development of Test Procedure:

Upon conclusive evidence that the local sand used in the Bradley pavement had played an active part in the failure, a laboratory study of this sand was undertaken to determine, if possible, the cause or causes of the trouble and the reason for the inconsistencies that had been noted not only in the locality under immediate investigation, but likewise in all of the coast counties from Monterey to and including the northwesterly part of Los Angeles County.

The first step was to make up mortar bars using the same brand cement that had been used in the Bradley pavement and the local (Oro Fino) and imported (Coyote) fine aggregates. Some of these bars were subjected to continuous wetting, some to continuous dry exposure and some to alternate wetting and drying.

Under none of these curing methods was any excessive expansion observable, even up to more than 1 yr., regardless of the source or nature of the sand.

However, upon removing the cover from a 2 by 4-in. mortar cylinder which had been cast and retained in a tin container for one year, the specimen was observed to be covered with blotches fringed with a white efflorescence (subsequently analyzed as sodium carbonate), and in a short time the entire specimen became covered with cracks similar to the cracks noted in pavements and other structures in the area under investigation.

A series of tests was then started on mortar bars cured in sealed containers, as well as in water. Experience with thousands of specimens, subsequently fabricated, has invariably checked the results noted in the case of the 2 by 4-in. mortar specimen. Bars fabricated with reactive sands and high-alkali cements and stored in sealed containers with a small amount of water in the bottom to maintain optimum humidity have invariably followed the same excessive expansion pattern.

Cement:

When the Bradley pavement failure was under investigation it was observed that other concrete structures in this area in which the same type of local sand but a different cement had been used developed no distress over long periods of time. This fact gave rise to the suspicion that the expansion resulted from a reaction between certain ingredients in the aggregate and some ingredients in the cement. Alkali in the cement in the form of the sodium and potassium oxides was suspected, and, as the cement used on the Bradley job contained approximately 1.14 per cent total alkali as Na_2O, the tests were repeated with another cement low in alkali (0.45 per cent), with definite results in that there was little, if any, expansion with the low-alkali cement in combination with the local (Oro Fino) sand which had shown so much expansion with the 1.14 per cent alkali cement. The tests have been repeated many times, not only with the two aforementioned cements, but with other California cements of low to high alkali content and sands from within and without the coast area.

Within the area under investigation, all of the aggregates from commercial sources, with one possible exception, contain appreciable percentages of shale and chert. All sands in the coast area relatively high in shale and chert develop expansions in the sealed container test in a few weeks (particularly when cured at 100 F.) in a magnitude roughly proportioned to the alkali content of the cement. Negligible short-time expansion is observed with cements containing less than 0.6 per cent alkali and early high expansion in the case of the higher alkali cements, roughly in proportion to the alkali content. Some inconsistencies have been noted but not of such magnitude that they cannot be accounted for by variations in the percentage of reactive minerals in the aggregate.

It is obvious, therefore, that in the presence of certain minerals an otherwise sound but high-alkali cement is a contributing factor to the excessive expansion and subsequent failure of a concrete structure.

Aggregates:

The coast aggregates included in the early California studies consist of mineral particles predominantly granitic in character with some quartz-feldspar, sandstone, impure limestone, shale, and chert. The granite, quartz-feldspar, and sandstone usually comprise more than 90 per cent of the total, the opal-containing limestones, shales, and cherts comprising the remaining 5 to 10 per cent. None of the adverse reactions have been traced to the first group. The opaline rocks in the second group, however, are definitely reactive under appropriate exposure conditions.

Undoubtedly opaline shales and cherts are acted upon by the caustic reaction products of cement hydration with the formation of sodium or potassium silica gel. Extensive tests show that many types of shale and chert found in the gravel deposits in the area under discussion are readily dissolved and disintegrated by soaking in sodium and potassium hydroxide solutions.

The probable rôle of the hydrous silicates was fully discussed in the earlier paper.[3] It was pointed out that a sodium silicate gel was at the base of most, if not all, of the popouts and was found throughout broken sections of concrete as well as exuding on the surface through pores and cracks.

Other Tests:

As soon as the cause of the Bradley and other failures in the same general area became known and a method of test had been developed to reproduce field experience in the laboratory, tests of aggregates from all but one of the commercial sources in southern Monterey, San Luis Obispo, Santa Barbara, and Ventura Counties have invariably followed the same pattern: namely, excessive expansion and cracking with a high-alkali cement and negligible expansion with the lower alkali cements, thereby paralleling field experience, good as well as bad—good with the low-alkali cements and the local aggregates, bad with cements containing in excess of 0.6 per cent alkali.

The correlation between the laboratory and field results is too significant to be disregarded.

Other Types of Aggregates:

All of the failures so far described have resulted from the presence of opal as the reactive mineral in the opaline cherts which contaminate practically all gravel deposits throughout the area.

When the U. S. Bureau of Reclamation determined that the cause of similar trouble at the Parker Dam on the Colorado River could be attributed to a similar reaction, it was found that the reactive aggregate was an andesitic rock. Here again, it is understood that the Bureau has invariably been able to reproduce in the laboratory, by the sealed-container test, the field experience with the same aggregates.

Expanding the field of study in California to other areas of the state, similar failures were observed in the vicinity of Fresno whenever aggregates from the San Joaquin River had been used in combination with high-alkali cements. Further north in the northeastern and High Sierra sections of the state where certain types of andesites are abundant, similar conditions have been observed. In every case it was possible subsequently to reproduce in the laboratory the same results with the same local aggregates, provided sufficient of the reactive minerals or rocks were present. Companion structures in the same areas, in which proved nonreactive aggregates but the same cements had been used, have never developed excessive expansion either in the field or laboratory and in other cases where the known reactive aggregate had been used with a low-alkali cement, but no distress had developed, subsequent laboratory tests with the identical reactive aggregates and low-alkali cements developed no undue expansion.

In order to become informed of the potential reactive properties of the aggregates from all sections of the state, tests have been made which included concrete sands from all commercially operated deposits without prior inspection of structures in which these sands had been used. The tests indicated that several of the sands hitherto unsuspected were potentially reactive. A subsequent inspection of structures in which these same aggregates had been used with a high-alkali cement disclosed excessive cracking typical of the aggregate-cement reaction.

In one case, the sand from a coast area known to contain reactive opaline cherts failed to develop excessive expansion. A subsequent petrographic study determined that the unreactive sand was free of the reactive type of shale and chert. No failures have ever been noted where this sand was used, thereby confirming laboratory tests. A few years ago several small areas of a recently (1936) constructed pavement, where a low-alkali cement had been used with a reactive aggregate, developed the typical expansion cracking noted with the same aggregates and high-alkali cements. A study of the resident engineer's diary disclosed that the contractor had used small quantities of high-early-strength high-alkali cement of a different brand on the approaches to several bridges and other locations where it was desired to open to traffic at an early date. The failed areas conformed in every instance to areas in which the high-alkali cement had been used. The balance of the project, in which the low-alkali cement was used with the same reactive aggregates, has developed no expansion cracks to date (12 yr. after construction). Laboratory tests with the same low-alkali cement and reactive aggregates have never produced excessive expansion, whereas similar laboratory tests with the same aggregates, but with the high-alkali brand cement which caused a failure on the above project, have always expanded excessively in the sealed container test.

This discussion relates entirely to test procedure where fine aggregates only are involved and the mortar bar specimens do not exceed 2 by 2 in. in cross-section. Very few tests on larger size concrete specimens containing coarse aggregate have been made, but such tests as have been made indicate that the sealed-container procedure is not necessary with the larger concrete specimens but that if there is potential reactivity excessive expansion and cracking will develop, provided sufficient but not excessive moisture is present to develop the reactive action.

CONCLUSION

No effort has been made in this discussion to list or describe a large number of projects, but rather to recite enough widely distributed incidents which, combined with the general observations cited, have been sufficient to demonstrate conclusively a close correlation between field performance and the results obtained in the sealed-container test.

For a complete description of similar studies and observations by other agencies, the reader is referred to the excellent bibliography in the paper on Cement-Aggregate Reaction in Concrete by Duncan McConnell, *et al.*, published in the *Journal* of the American Concrete Institure.[5]

---

[5] Duncan McConnell, Richard E. Mielenz, William Y. Holland, and Kenneth T. Green, "Cement-Aggregate Reaction in Concrete," *Journal*, Am. Concrete Inst., Vol 19, No. 2, October, 1947; *Proceedings*, Vol. 44, p. 93.

DISCUSSION

MR. W. C. HANNA[1] (*presented in written form*).—It is a real pleasure to comment on this paper. Many of us have followed closely the studies of the alkali-aggregate reactions which were so ably initiated by Mr. Stanton and his associates. Throughout his work he has never deviated from his objective of sound concrete for use in the State of California. We are also pleased to note his sense of fairness in acknowledging the contributions of other outstanding laboratories in the study of this involved problem.

Mr. Stanton makes a strong point for the indicated correlation between the Sealed Container - Mortar Bar Test and actual field conditions. Data of this kind are valuable as they can be a guide to future requirements for new concrete structures such as bridges and highways.

As to the details of the test, our experience has indicated the desirability of a completely sealed container. However, Mr. Stanton has tests to indicate that fairly tight containers are satisfactory.

We note that Mr. Stanton has to some extent associated the brand of cement with its alkali content. We would avoid this as we know that a given plant can have variable alkali in its cement through the years. We have tabulated the maxima and minima for the sodium oxide of various cements on which we have kept records for the past seven years.

Brand	Actual Analysis, per cent			Maximum, per cent		Minimum, per cent	
	Na_2O	K_2O	Total Alkali as Na_2O	Na_2O	K_2O	Na_2O	K_2O
A	0.57	0.40	0.83	0.57	0.40		
A	0.16	0.06	0.20			0.16	0.06
A	0.19	0.05	0.22			0.19	0.05
B	0.83	0.38	1.08	0.83	0.38		
B	0.51	0.39	0.77			0.51	0.39
B	0.77	0.45	1.07	0.77	0.45		
B	0.72	0.24	0.88			0.72	0.24
C	0.51	0.31	0.71	0.51	0.31		
C	0.26	0.24	0.42			0.26	0.24
C	0.39	0.21	0.53			0.39	0.21
D	0.51	0.50	0.84	0.51	0.50		
D	0.14	0.48	0.46			0.14	0.48
D	0.22	0.60	0.62	0.22	0.60		
D	0.24	0.31	0.48			0.24	0.31

A study of this table shows cement of brand A having a total calculated sodium oxide range from 0.20 per cent to 0.83 per cent, brand B a range from 0.77 per cent to 1.08 per cent, brand C from 0.42 per cent to 0.71 per cent, and brand D from 0.48 per cent to 0.84 per cent. We know of other authoritative analyses that would extend the ranges of these extremes still further.

Mr. Stanton's work is almost completely directed at the specific alkali-aggregate reaction. In the interest of insuring good concrete we like to look at the problem in its broadest sense. This involves a complete study of *each* component of the concrete mix as well as a survey of any chemical or physical changes which may occur during the placing of the concrete or its subsequent treatment.

We again wish to thank Mr. Stanton for his excellent summation of his alkali-aggregate experience. Work of this kind should encourage all of us to continue our efforts toward finding out what is the true mechanism of the alkali-aggregate reaction.

---

[1] Chief Chemist and Chemical Engineer, California Portland Cement Co., Colton, Calif.

MR. BAILEY TREMPER[2] (*presented in written form*).—The author shows in a convincing manner that a high degree of correlation exists between the Sealed Container - Mortar Bar Test and field experience with reactive aggregates. Examples are cited of distress in structures when high-alkali cement was used in combination with aggregates that were shown to be reactive in the mortar bar test. Of equal importance is the report that no abnormal expansion was found when low-alkali cement was used with such aggregates. Instances of pavements and structures of ages up to 18 yr. that are still in excellent condition point unmistakably to the conclusion that a satisfactory method of avoiding distress with reactive aggregates is to use low-alkali cement. Satisfactory results have been noted with two distinct types of reactive aggregate, namely, that containing opal and that containing andesite. Since these two groups comprise the bulk of the reported types of reactive aggregate, it is particularly reassuring to know that failure to obtain significant expansion with low-alkali cement in the laboratory mortar-bar expansion test is good evidence that field experience will be satisfactory.

MR. HARMON S. MEISSNER.[3]—Mr. Tremper in his discussion touched upon the same thing that I desire to comment on, namely, that Mr. Stanton now has 12 years' experience with cement containing less than 0.6 per cent total alkalies when used with reactive aggregates. He states that, so far, those structures are in excellent condition.

The Bureau of Reclamation's first use of a low-alkali cement was in the construction of a power plant at Parker Dam. Those who have studied this problem, know that Parker Dam was our first experience with this trouble. The power plant was built some two years after the dam, and at that time the dam was already cracking. We discovered what we had there and naturally called for a low-alkali cement in building the power plant. The power plant has been in existence for eight years and is in perfect shape, although some of the foundations built at the time the dam was built are cracking badly. So now we have 8 years, and in the case of Stanton 12 years, experience with low-alkali cement, finding it entirely satisfactory with highly reactive aggregates.

SESSION CHAIRMAN R. E. DAVIS.[4]—I wonder whether Mr. Hanna would care to comment on Mr. Stanton's conclusion that with less than 0.6 per cent alkali there would be no abnormal expansion in concrete.

MR. HANNA.—My contention was that the alkali content of any brand of cement is not always the same. It might be very possible that some of the good concrete structures were made with high-alkali cements. That is what I wished to imply. You might get good structures with either high alkali or low-alkali cements, depending on the entire chemical reaction of all the aggregates and everything else that is in the concrete. Looking back at structures as old as some of these that are passed upon as being good structures with low-alkali cement, I do not believe anybody can be certain whether the cement was low in alkali or high in alkali.

MR. DAVIS.—What might happen with a low-alkali cement if a portion of the sand were high in alkali?

MR. HANNA.—I think it is very probable that the alkali in the aggregates often far exceeds the alkali in the cement, if you consider Clarke's famous book, "Data of Geochemistry," for example, and see the composition of the earth's crust and the materials that probably would be considered to be good aggregates. You would find that the aggregates could have as much as about 3½ per cent sodium oxide and about 3 per cent potassium oxide. Those are used in concrete, and when you have concrete with a mixture of cement you get an alkali reaction. Calcium, sodium, potassium—whatever might be in the mixture—could possibly react, some beneficially and some otherwise.

---

[2] Materials and Research Engineer, Washington Department of Highways, Olympia, Wash.

[3] Research Engineer, Bureau of Reclamation, Denver, Colo.

[4] Director, Engineering Materials Lab., University of California, Berkeley, Calif.

At one time in a discussion of Mr. Stanton's paper, several years ago, I submitted a table showing expansion tests on about 100 samples of cement running from very low alkali to very high alkali. At a period of about four years or less every sample showed high expansion, regardless of the alkali, indicating that low-alkali cement was not a cure for bad aggregates or reactive aggregates in every case. All of these mortars contained a portion of a known bad aggregate.

In the closure to the paper, as I recall it, the point was made that neutral sand showed no expansion or very little—a few hundredths of one per cent—with the same combination, and with Ottawa sand the same thing. Either high alkali or low alkali cements did not work with the other combination of aggregates because the neutral sand was high in alkali. So the supposition is that sand with 3 or 4 per cent of alkali in it, whatever the earth's crust is, is no good. However, such is not the case, because as I have just said, many of the best aggregates all over the country are high in alkali.

MR. DAVIS.—I think Mr. Hanna has brought out a very good point, that under certain conditions a low-alkali cement may not prevent abnormal expansion.

MR. STANTON WALKER.[5]—I want to ask a very simple question, and I am sure there are many in the audience who can answer it. Mr. Hanna has referred to the alkali content of aggregates, and he has referred to sodium and potassium oxides of the order of 3½ or 4 per cent. I am wondering if these sodium and potassium oxides occur as oxides and are available to go in solution as the oxides are in cement? So, I am wondering if an oxide analysis of aggregates showing alkalies is of significance in the same way that an oxide analysis of cement would be?

MR. HANNA.—Absolutely not, because you have a great many combinations of silica and both sodium and potassium. You have sodium silicates, potassium silicates, and other very complex combinations in the earth's crust. All you have to take is some of those books, like Clarke's "Data of Geochemistry" and see what combinations you can get; they are just enormous and you could expect variable results.

MR. THOMAS E. STANTON (*author's closure by letter*).—Mr. Hanna has presented some very interesting data showing the variation over a period of years of the alkali content in the same brand of cement. From these data he deduces the theory that the potential alkali content of a cement should not be associated with the brand. The association of the alkali content with the brand was purely coincidental and not of any particular significance, except that the source of raw materials and manufacturing processes at a given mill are usually uniform over periods of time and a knowledge of the normal characteristics of a given brand of cement are an excellent guide to the user. In the final analysis, of course, the amount of alkali in each lot of cement is the significant factor and not the brand.

---

[5] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

Mr. Hanna makes the contention that under certain conditions a low-alkali cement may not prevent abnormal expansion. He evidently has in mind the high expansions which he observed in a series of tests conducted by him, using some low-alkali cements in combination with a local sand (Colton Plaster) which is high in alkali. It was determined by us that this local sand contains approximately 6 per cent total alkalies. Assuming that the alkalies are at the root of the trouble, it is reasonable to conclude that the excessive expansion observed by Mr. Hanna in a low-alkali cement-high-alkali aggregate containing mineral particles readily attacked by alkali, such as opaline chert was due at least in part to the alkalies in the aggregate. If such is the case it would appear that the alkalies in the aggregate in question were present in some form available for action.

The above discussion, however, is not in any way related to the topic of the paper, namely, the "Correlation of Laboratory Tests with Field Experience." The data simply tends to confirm the conclusion that alkali is one of the factors at the root of the trouble whether occurring in the aggregate or cement. At the same time, the bulk of the evidence to date indicates that the most likely occurrence of alkali available for the reaction will be found in the cement and not in the aggregate.

While it is true that considerable mystery still surrounds certain manifestations of the subject, we have had no occasion to change the conclusions reached from the start (1939), namely, that concrete failures of the type reported can be traced directly to some mineral in the aggregate which is attacked by some constituent of the cement with the resultant formation of a sodium or potassium silica gel which, in an unconfined state, swells to many times the original volume of the components of the gel and which before relief can be afforded, exerts an enormous pressure which ruptures the concrete and that the probability that this condition will be experienced in the field can be predicted from the results of laboratory tests of the nature described.

The process by which this disruptive force develops is discussed by W. C. Hansen, now with the Universal Atlas Cement Co., in a paper published by the American Concrete Institute.[6]

It has also been established that the reactive mineral in the aggregate usually occurs in the form of a silica readily soluble in sodium or potassium hydroxide, such as opal and the glassy matrix of some volcanic rocks, such as the andesites. Furthermore, it has been definitely established through longtime observation covering at least 25 yr., that the destructive action, with the aggregates found in California, has occurred only when the combined Na_2O and K_2O of the cement exceeded 0.6 per cent.

It is true that excessive expansions have been observed in time in laboratory tests with lower alkali content cements. However, the manifestation of excessive reaction is slow in developing, and no evidence has been observed that any ultimate abnormal reaction in the accelerated laboratory test with low-alkali cements will be reproduced under normal field exposure conditions, except possibly with some special combination not yet encountered.

The fact, however, that in time excessive expansion may occur with one cement of relatively low-alkali content and no excessive expansion with another brand cement, but the same alkali content, indicates that the manner of occurrence of the alkalies may influence the results and, in my opinion, a study of the conditions under which this may occur offers one of the most fertile fields for further studies of the problem, to the end that we may develop a more discriminating specification than a uniform limitation on the alkali content regardless of the nature of its occurrence.

---

[6] W. C. Hansen, "Studies Relating to the Mechanism by Which the Alkali-Aggregate Reaction Produces Expansion in Concrete," *Journal*, Am. Concrete Inst., Vol. 15, No. 3, January, 1944; *Proceedings*, Vol. 40, p. 213.

CORRELATION OF LABORATORY TESTS WITH FIELD EXPERIENCE IN ALKALI-AGGREGATE REACTION*

By Bailey Tremper[1]

Synopsis

A number of kinds of laboratory tests show sand and gravel from Cowlitz River, Washington, to be detrimentally reactive with high-alkali cement. Mortar bar expansion tests and freezing-and-thawing tests also indicate good durability when low-alkali cement is used with these aggregates. The present condition of sixteen concrete bridges, constructed with these aggregates and ranging in age from 11 to 23 yr., is described. These observations indicate a high degree of correlation between laboratory tests and field experience.

Studies of the reactivity of sand and gravel from the Cowlitz River in Washington have been made in a number of laboratories following publication in 1941 of a report (**1**)[2] describing abnormal expansion and cracking in structures containing these aggregates when used with high-alkali cement. All of the reports of such tests that have come to the author's attention have been in agreement in that they indicate the aggregates, either sand or gravel, to be reactive with high-alkali cement.

The test methods reported by various investigators include petrographic examination of the aggregate particles, petrographic examination of concrete containing the aggregate, observation of beads and exudations on cores from affected structures, etching tests of gravel particles, mortar bar expansion tests, freezing-and-thawing tests, and chemical tests for reactivity of aggregates in cement alkalies.

The last-named test, published by Mielenz, Greene, and Benton (**2**) is of particular interest because it can be completed in an elapsed time of two or three days and because it yields numerical values that are used to characterize the aggregate, either as innocuous or deleterious. The report of these investigators places Cowlitz River sand and gravel in the deleterious group, a finding that agrees with results of their mortar bar expansion tests.

Tests made in the laboratory of the Washington Department of Highways confirm the results of Mielenz, Greene, and Benton. More extensive data, however, have been developed through physical tests of mortar and concrete. Freezing-and-thawing tests of concrete containing Cowlitz River aggregates have indicated a lack of resistance when high-alkali cements have been used but good durability when the cements were low in alkali (**3**).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Materials and Research Engineer, State of Washington, Department of Highways, Olympia, Washington.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1070.

Mortar expansion bars containing these aggregates have been in sealed storage at 70 F. for periods up to 7 yr. and results are shown in Fig. 1. These tests have been conducted in a manner similar to that described in the report of the Working Committee on the Effect of Alkalies in Portland Cement of A.S.T.M. Committee C-1 (4), except that the mix used was 1 part cement to 2 parts sand, by weight. These long-time tests are of interest because they show no increase in expansion with time when low-alkali cements are used, but continuously increasing expansion throughout the storage period when high-alkali cements are used. There is no indication that expansion will cease in the near future. The expansion at 1 yr. is not sufficiently great to indicate definitely that the aggregates are reactive. Although the rate of expansion has decreased somewhat after the third year, it is to be noted that, with the high-alkali cements, the total expansion at the end of 6 yr. is from five to six times that at 1 yr. At the end of 7 yr. there is little doubt that cement with an alkali content as low as 0.77 per cent reacts detrimentally.

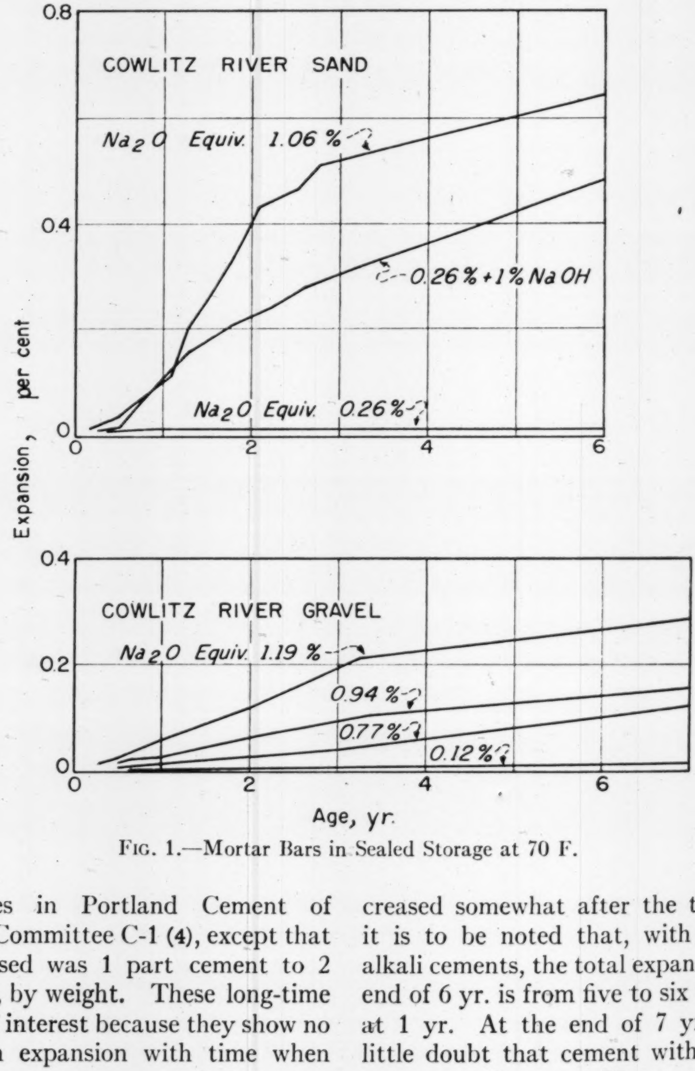


FIG. 1.—Mortar Bars in Sealed Storage at 70 F.

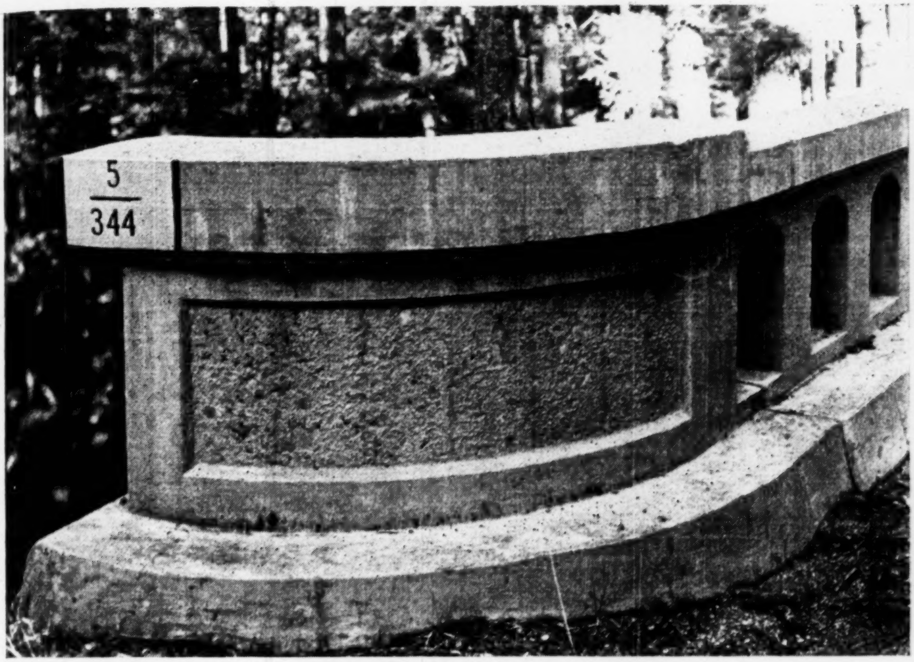


FIG. 2.—Concrete Containing Low-Alkali Cement.

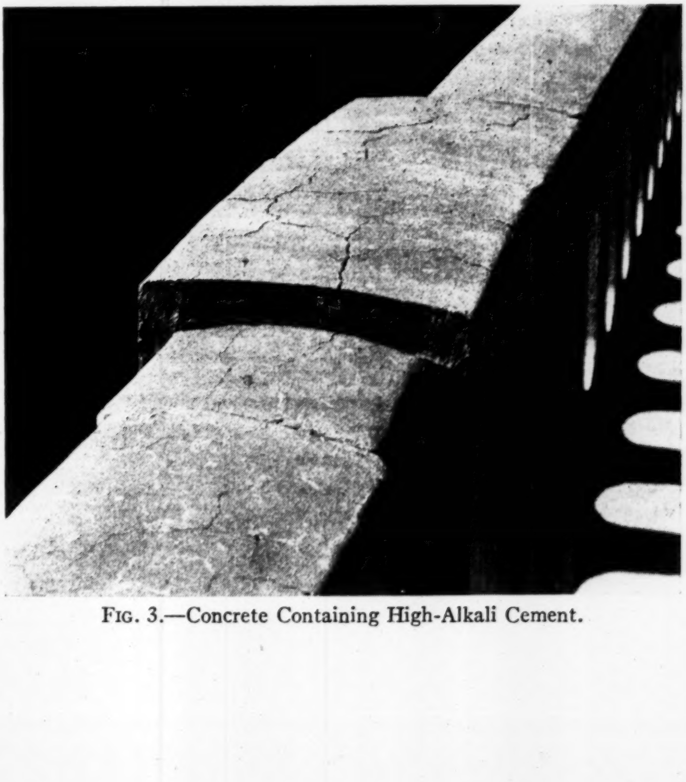


FIG. 3.—Concrete Containing High-Alkali Cement.

With this background of laboratory test results in mind, it is of value to inquire into their significance with respect to performance in service as demonstrated by a group of structures containing Cowlitz River aggregate and both high- and low-alkali cements.

In the low-alkali group there are ten bridges ranging in age from 11 to 23 yr. Each is in excellent condition today. Part were constructed with cement estimated to have had an alkali content of 0.50 to 0.55 per cent (Na_2O equivalent). In others, the alkali content of the cement probably was of the order of 0.25 per cent. Figure 2 is a view of the handrail of one of these bridges constructed 19 years ago. Note the sharpness of line and complete freedom from cracking.

In contrast to this condition, note the appearance in Fig. 3 of the handrail of a nearby bridge constructed a few years later with a high-alkali cement (Na_2O equivalent estimated to be about 1.00 per cent). Cracking and warping are prominent in this view, but abnormal expansion is not well illustrated. Openings ¼ in. wide were provided at 12-ft. intervals in the handrails. Nearly all of these are now closed tight and in some the pressure of expansion has been great enough to cause spalling. The expansion of these members has been about 0.2 per cent, a value not out of line with laboratory sealed storage tests.

There are six structures containing high-alkali cement in this vicinity. The general condition of the handrails is not greatly different from that shown in Fig. 3. A smaller amount of cracking occurs in the substructures, but it is present nearly everywhere. The process of deterioration is continuing. Cracks have rendered the concrete susceptible to the action of freezing and thawing. Thin, exposed members such as handrails and curbs are now showing signs of disintegration from this cause.

The service records of these structures, embodying, as they do, examples of both high- and low-alkali cements, indicate a high degree of correlation with the results of laboratory tests. All of the tests mentioned in the opening paragraphs of this paper indicate that the aggregate is reactive with high-alkali cement. Among these, only the freezing-and-thawing test and the mortar bar expansion test indicate the observed fact that when used with low-alkali cement, a high degree of permanence can be expected from concrete made with Cowlitz River aggregates even though it is exposed to severe weathering conditions.

REFERENCES

(1) Bailey Tremper, "Evidence in Washington of Deterioration of Concrete Through Reactions between Aggregates and High-Alkali Cements," *Journal*, Am. Concrete Inst., June, 1941; *Proceedings*, Am. Concrete Inst. Vol. 37, p. 673.

(2) R. C. Mielenz, K. T. Greene, and E. J. Benton, "Chemical Test for Reactivity of Aggregates with Cement Alkalies, Chemical Processes in Cement Aggregate Reaction," *Journal*, Am. Concrete Inst., November, 1947; *Proceedings*, Am. Concrete Inst., Vol. 44, p. 193.

(3) Bailey Tremper, "The Effect of Alkalies in Portland Cement on the Durability of Concrete," *Journal*, Am. Concrete Inst., November, 1944; *Proceedings*, Am. Concrete Inst., Vol. 41, p. 89.

(4) "Effect of Alkalies in Portland Cement on Durability of Concrete," Report of Working Committee of A.S.T.M. Committee C-1 on Cement, ASTM BULLETIN, No. 142, October, 1946, p. 28.

TESTS USED BY THE BUREAU OF RECLAMATION FOR IDENTIFYING REACTIVE CONCRETE AGGREGATES*

BY RICHARD C. MIELENZ[1] AND LESLIE P. WITTE[1]

SYNOPSIS

Research and investigations under way since 1941 have evolved five techniques which together will reliably evaluate alkali reactivity of aggregates in minimum time. These techniques are: (1) Petrographic examination, (2) a chemical test with NaOH solution, (3) a test involving length change measurement of moist-stored mortar specimens, (4) a test involving length change measurement of concrete beams subjected to alternate wetting and drying with simultaneous heating and cooling, and (5) examination of concrete structures in which the aggregate was used. Determination of reactivity by the petrographic examination and chemical test can be completed in three work days. The mortar and concrete tests offer a more critical evaluation of deleteriousness, but the determination may require several months of testing. Examination of concrete structures to establish the deleterious or innocuous character of the aggregate is meaningful only if conducted by an experienced engineer to whom data pertinent to the fabrication and conditions of service of the concrete are available.

The program comprising four tests and examination of existing structures is recommended for use wherever alkali reactivity of aggregates is suspected. Details of the procedures and necessary apparatus are described in appendices to this paper.

Following the discovery by T. E. Stanton(**1**, **2**)[2] that expansive deterioration of concrete can arise from chemical interaction between cement alkalies and certain aggregate constituents, and the identification in 1940 of this reaction in a concrete at Parker Dam, California-Arizona (**3**), the Bureau of Reclamation undertook a program of tests and research to determine, among other things, means by which potential reactivity of aggregates could be evaluated quickly and reliably. The program entailed: (1) Petrographic investigation of aggregates whose deleteriousness was known; (2) development of a practicable chemical test of reactivity; (3) an extensive program of tests of concrete and mortar; and (4) investigation and examination in field and laboratory of concrete in structures. Effective prosecution of the program would have been impracticable without the cooperation of the many public and private agencies who supplied samples of aggregate and concrete with correlative service histories relating to affected structures in many parts of the United States.

From these investigations, five techniques have been evolved by which the potential deleteriousness of aggregates used in construction on projects of the Bureau of Reclamation is determined.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Head, Petrographic Laboratory, and Materials Engineer, respectively, Bureau of Reclamation, United States Department of the Interior, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1095.

TABLE I.—PETROGRAPHIC ANALYSIS OF COARSE AGGREGATE.
Enders Damsite, Frenchman-Cambridge Unit, Missouri Basin Project.

Rock Types	Size-Fraction, per cent by weight						Description of Rock Types	Classification
	No. 7763		No. 7766		No. 7843			
	1½ to ¾ in.	¾ to ⅜ in.	1½ to ¾ in.	¾ to ⅜ in.	1½ to ¾ in.	¾ to ⅜ in.		
Granites	40.8	64.1	52.7	58.5	37.0	69.4	Slightly weathered, coarse-grained	Innocuous
Weathered granites	13.2	6.4	12.1	4.5	11.7	3.1	Coarse-grained, slightly fractured	Innocuous
Deeply weathered granites	3.0	1.3	...	2.4	9.1	3.0	Coarse-grained, fractured, friable	Innocuous
Sandstones	21.2	13.7	23.6	16.5	23.0	13.7	Slightly weathered, coarse to fine	Innocuous
Weathered sandstones	2.9	0.7	...	1.6	4.1	4.3	Coarse- to fine-grained	Innocuous
Deeply weathered sandstones	1.7	0.2	...		1.2	...	Coarse- to fine-grained, friable	Innocuous
Quartz	12.4	9.4	2.9	7.1	11.6	4.7	Hard, smooth, poor bonding characteristics	Innocuous
Rhyolites	...	0.9		0.7	...	1.4	Slightly weathered, aphanitic, porphyritic	Deleterious
Rhyolite porphyries	1.6	1.6	8.7	1.7	...	...	Slightly weathered, fine-grained to aphanitic	Innocuous
Andesites				1.8	...	...	Slightly weathered, aphanitic, porphyritic	Deleterious
Andesite porphyries		0.9		1.1	...	...	Slightly weathered, fine-grained, porphyritic	Innocuous
Weathered andesite porphyries			...	1.3	...		Fine-grained, porphyritic	Innocuous
Cherts	3.2	0.8	...	2.8		0.4	Hard, chalcedonic	Deleterious
Conglomerate	...	...	...	...	2.3	...	Coarse- to fine-grained, friable, chalcedonic	Deleterious

PETROGRAPHIC EXAMINATION OF AGGREGATES

Since 1936 all aggregates to be used in construction on Bureau projects have been examined and analyzed petrographically to supplement determinations made by standard tests of quality. The petrographic examination is performed not only to determine the potential degree of reactivity of the aggregates but also to establish their physical quality (**4, 5**). However, in this discussion only the techniques and problems related to the determination of reactivity will be discussed. The procedure of the petrographic examination is described in detail in Appendix I.* Results of petrographic analyses of coarse and fine aggregate are summarized to indicate the identity and abundance of particles considered to be deleteriously reactive (Tables I and II).

Petrographic examination of aggregates to evaluate reactivity with the cement alkalies is essentially a visual inspection by which the presence and abundance of the particles containing reactive substances are determined.

---

* See p. 1096.

TABLE II.—PETROGRAPHIC ANALYSIS OF SAND.
Hoover Dam, Boulder Canyon Project, Nevada-Arizona
Sample No. P-1441B.

Rock and Mineral Types	Size-fraction—Proportion by Count of Particles, per cent				Classification
	No. 4 to No. 8	No. 8 to No. 16	No. 16 to No. 30	Nol 50 to No. 100	
Granitic rocks[a]	10.3	21.6	22.6	...	Innocuous
Gneisses and schists	9.8	6.3	12.2	...	Innocuous
Rhyolites	7.5	8.7	8.6	...	Deleterious
Rhyolite tuffs without opal	9.8	8.7	7.0	...	Deleterious
Rhyolite tuffs containing opal	4.6	1.2	0.9	...	Deleterious
Augite andesites	3.4	3.5	2.5	...	Deleterious
Deeply altered andesites	5.7	5.9	4.9	...	Deleterious
Olivine basalts	...	2.8	0.9	...	Innocuous
Sandstones	4.0	2.0	2.5	...	Innocuous
Limestones	20.1	11.4	7.9	3.4	Innocuous
Chalcedonic limestones	8.0	9.8	7.6	...	Deleterious
Opaline limestones	4.1	0.8	0.3	...	Deleterious
Chalcedonic chert	12.1	15.3	19.6	...	Deleterious
Opaline chert	0.6	2.0	2.5	...	Deleterious
Quartz	...	...	...	64.5	Innocuous
Potash feldspars	...	...	...	9.2	Innocuous
Plagioclase feldspars	...	...	...	4.5	Innocuous
Opal	...	...	...	0.2	Deleterious
Heavy minerals	...	...	...	2.7	Innocuous
Unidentified	...	...	...	15.5	...

[a] Includes some individual grains of quartz and feldspars.

From petrographic analysis of aggregates known to be innocuous or deleterious, as the case may be, as a result of laboratory and field investigations, the rocks and minerals participating in alkali-aggregate reaction in mortar and concrete have been identified. The known reactive substances are the silica minerals, opal, chalcedony, tridymite (6) and cristobalite (7); the zeolite, heulandite (and probably ptilolite); glassy to cryptocrystalline rhyolites, dacites and andesites and their tuffs; and at least certain phyllites. Any rock containing a significant proportion of reactive substances will be deleteriously reactive; thus, although pure limestones and dolomites are not deleteriously reactive, opaline or chalcedonic limestones and dolomites are related to deterioration of concrete as a result of alkali-aggregate reactivity. Similarly, normally innocuous sandstones, shales, granites, basalts, or other rocks can be deleteriously reactive if they are impregnated or coated with opal, chalcedony, or other reactive substances. For example, an olivine basalt investigated as a source of concrete aggregate for use at Chiflo Damsite on the Rio Grande River, New Mexico, was found to contain about 20 per cent by volume of a glass which is andesitic rather than basaltic in composition, having an index of refraction of 1.510. The deleteriousness predicted on the basis of the petrographic analysis was confirmed by high-alkali mortar bars which expanded 0.224 per cent linearly during 12 months of moist storage.

Based upon investigation of aggregates of known deleteriousness, the proportions of the various constituents necessary for production of distress in concrete can be estimated. In the Bureau of Reclamation procedure, aggregates containing more than 0.25 per cent by weight of opal; more than 5 per cent of chalcedony; or more than 3 per cent of glassy or cryptocrystalline acidic to intermediate volcanic rocks or tuffs are recommended for use only if cements low in alkalies are obtainable. These limits are probably conservative; furthermore, to the extent that the reactive ingredients are admixed with or enclosed within innocuous substances, the limits can be increased with safety. For example, opal may occur in isolated masses with innocuous materials, and hence will be less susceptible to attack. Chalcedonic cherts may contain considerable calcium carbonate so that the amount of reacting material is not truly indicated by the amount of chert in the aggregate. Also, otherwise highly reactive volcanic rock may be only slightly deleterious or even innocuous if the original glass is completely recrystallized as a result of geologic processes.

The petrographic analysis can be performed quickly and thus affords a practical means by which those materials most urgently requiring exhaustive tests can be selected. Depending upon the lithologic complexity of the aggregate, the petrographic examination requires one-half to two man-days for completion.

CHEMICAL TESTING OF AGGREGATES

As a result of intensive research, a practicable chemical test to measure potential alkali reactivity of aggregates has been developed (8). The procedure has been applied to 101 sands, gravels, minerals, and rocks whose degree of deleteriousness is established by tests of mortar and concrete, reliable service histories, or petrographic examination (Fig. 1). The test results correlate well with the amount and rate of expansion of moist-stored mortars containing high-alkali cement.

The test involves immersion of a sample of aggregate crushed to the No. 50 to No. 100 size in a 1 N solution of NaOH solution at 80 C. for 24 hr. At completion of the test, the solution is filtered off and analyzed for dissolved silica, and the reduction in alkalinity of the solution is determined by titration to the phenolphthalein end-point. Details of the procedure are described in Appendix II.*

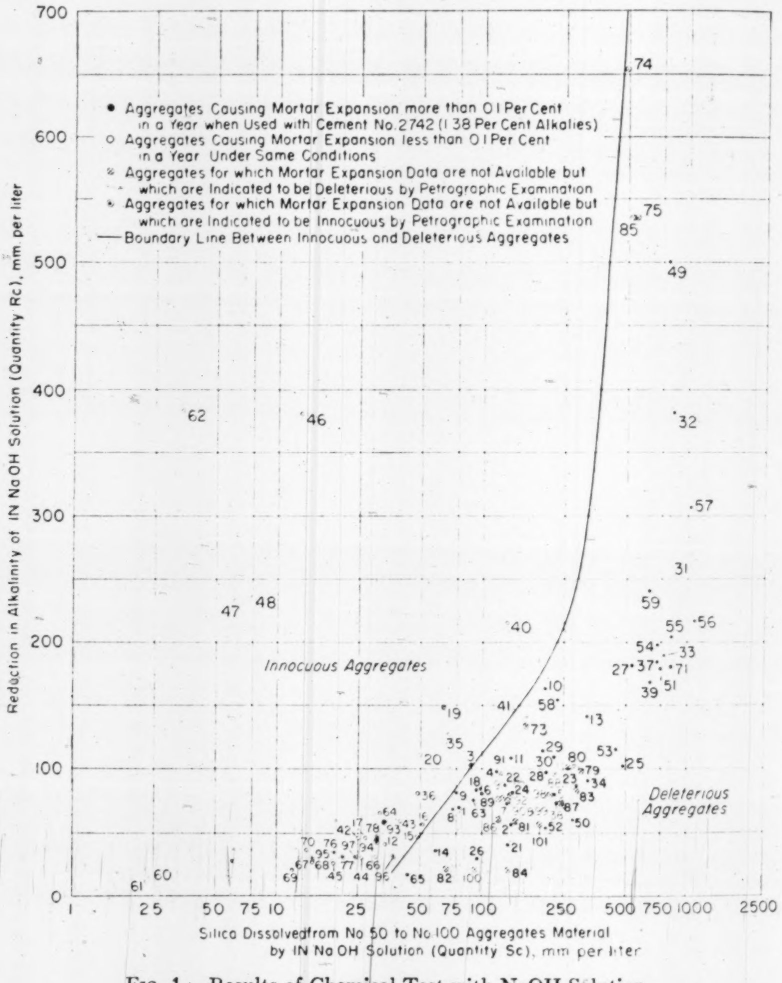


FIG. 1.—Results of Chemical Test with NaOH Solution.
Each Data Point Represents Average of 2 or 3 Determinations.

Deleterious materials usually are indicated by a ratio of $S_c:R_c$ greater than 1, where S_c expresses the concentration of dissolved silica in millimoles per liter and R_c expresses the reduction in titratable hydroxyl ion in millimoles per liter. In this test the determined concentration of silica in solution at completion of the test ranged from 2.3 to 1100 millimoles per liter (0.14 to 66 g. per liter) or from 0.01 to 6.60 per cent by weight of the tested sample. Reduction in alkalinity of the NaOH solution ranged from 12 to 678 millimoles per liter. For deleterious materials the magnitude of the reduction in alkalinity tends to vary inversely with the magnitude of the so-called "pessimum"[3] proportion of the aggregate.

---

* See p. 1097.

Preparation of an aggregate sample for testing, operation of the test, and the chemical analyses can be completed in three work days and involve about six man-hours of direct labor.

MORTAR EXPANSION TEST

Following the conference on "Alkalies in Cement" held in Denver in February, 1941 (**10**), the Bureau of Reclamation initiated an extensive program of tests and research to investigate the causes and controls of alkali-aggregate reaction in concrete and mortar. This program included investigation of the influence of the kind, amount, and gradation of the aggregate; cement content; size of specimen; conditions of curing and storage; and other factors. The results of many of these determinations have been summarized previously (**11**).

Also included in this initial program was a procedure involving the fabrication and periodic observation of mortar bars containing natural aggregates and high- and low-alkali cements. At monthly intervals for two years and, where desirable, less frequently to later ages, the length change and dynamic modulus of elasticity of the specimens were measured. Incorporated in this procedure were samples of aggregates upon which field service records were available, including many known to contribute to the deterioration of concrete through alkali-aggregate reaction. Sufficient data have now been obtained to correlate adequately the expansion of the mortar bars containing high-alkali cement with deterioration to be expected in field structures if the same aggregates are used with high-alkali cements.

This program of mortar tests designed primarily to determine the reactivity of aggregates previously used and proposed for use in field structures, was based on fabrication and testing of specimens 2 by 2 by 10 in. in size[4], composed of fine aggregate and cement in the ratio two parts to one by weight. The basic program required use of one high-alkali cement (No. 2742, 1.30 per cent Na_2O and 0.12 per cent K_2O) and one low-alkali cement (No. 2735, 0.04 per cent Na_2O and 0.14 per cent K_2O), but, where considered desirable, other cements of intermediate alkali content were used also. Cast in triplicate, the specimens were cured 7 days in the fog room, then stored at 100 F. in sealed cans in the presence of, but not in contact with, water. The sand was graded 19 per cent by weight each of the sizes No. 4 to No. 8, No. 8 to No. 16, No. 16 to No. 30, No. 30 to No. 50, and No. 50 to No. 100, with 5 per cent of material passing the No. 100 screen. Gravel and quarried rock to be used as coarse aggregate only was crushed and graded 20 per cent each of the five sizes[5]. Details of procedure are outlined in Appendix III.* The procedure is the same as that initiated in 1941, except that the size of the specimens has been reduced to 1 by 1 by 10 in. to facilitate handling.

In conjunction with this mortar program, a similar series of 1 by 1 by 10 in. mortar specimens containing selected rocks and minerals as aggregate was prepared, particularly to facilitate the correlation of expansivity of mortar with the lithologic composition of the aggregate. Data obtained from this series have enabled the Bureau's petrographers to predict with considerable reliability the potential reactivity of new aggregates proposed for use in construction.

---

[3] A word used by T. E. Stanton (**9**) to designate the proportion of reactive substances in an aggregate at which maximum mortar expansion occurs.

[4] The specimens are 2 by 2 by 11¼ in. in over-all dimensions, the distance between the base of the terminal reference points being 10 in.

[5] These gradings were selected so that data derived from this program could be correlated with results of other tests of mortar and concrete in which these same gradings were used. These latter tests were devised specifically to investigate the effects of successive replacement of innocuous aggregate by reactive aggregate in individual size fractions.

* See p. 1098.

Progress of alkali-aggregate reaction in mortar is indicated by progressive expansion of inordinate magnitude, and, at early ages, a decline in dynamic modulus of elasticity. Severe distress is accompanied by supplementary phenomena, such as cracking, exudation of alkalic silica gel from cracks, formation of popouts, and warping (Fig. 2). Microscopical examination may reveal the effects of the reaction prior to the development of the readily apparent surface features.

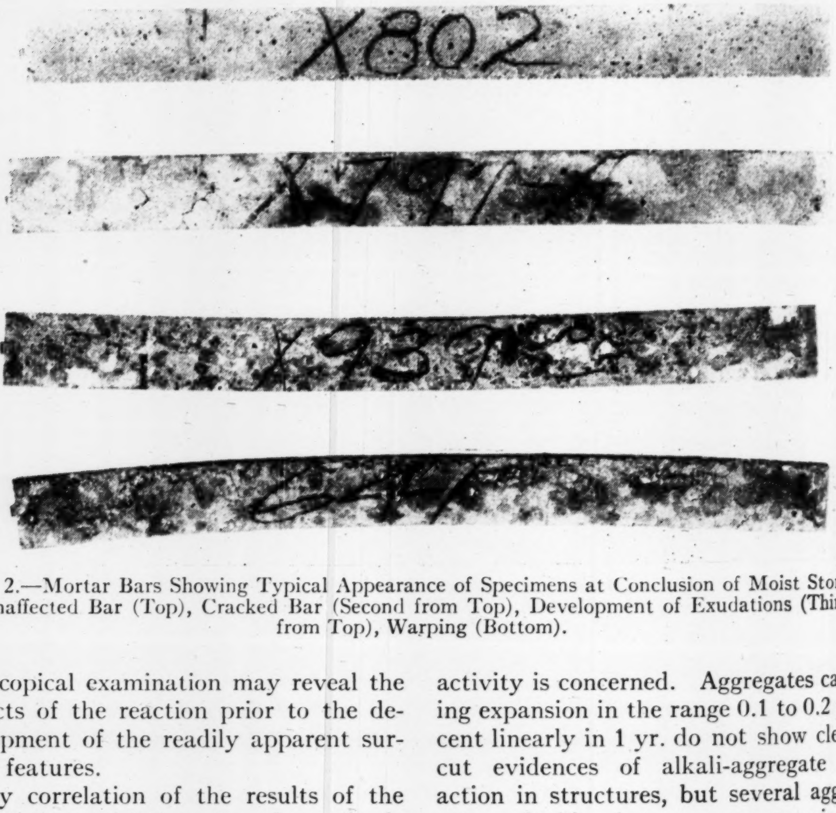


FIG. 2.—Mortar Bars Showing Typical Appearance of Specimens at Conclusion of Moist Storage Unaffected Bar (Top), Cracked Bar (Second from Top), Development of Exudations (Third from Top), Warping (Bottom).

By correlation of the results of the mortar tests with the rate and magnitude of deterioration due to alkali-aggregate reaction where the tested aggregate was used in field construction with high-alkali cement, it is established that aggregates causing a mortar expansion in excess of 0.2 per cent linearly in 1 yr. in this test with high-alkali cement No. 2742 (1.30 per cent Na_2O and 0.12 per cent K_2O) should be expected to cause rapid, intense, and readily identifiable expansive deterioration of concrete if used with a high-alkali cement in exposed situations. Aggregates causing a mortar expansion less than 0.1 per cent linearly in 1 yr., if used with the high-alkali cement, have been found to be innocuous so far as alkali-aggregate reactivity is concerned. Aggregates causing expansion in the range 0.1 to 0.2 per cent linearly in 1 yr. do not show clear-cut evidences of alkali-aggregate reaction in structures, but several aggregates of this character are associated with concrete deterioration whose cause is conjectural. Consequently, aggregates causing mortar expansion in excess of 0.1 per cent linearly in 1 yr. should be used only with cements low in alkalies. Usually, aggregate causing mortar expansion of 0.1 per cent or more at 6 months must be regarded as deleterious, whereas those causing expansions in excess of 0.04 per cent should be regarded with suspicion.

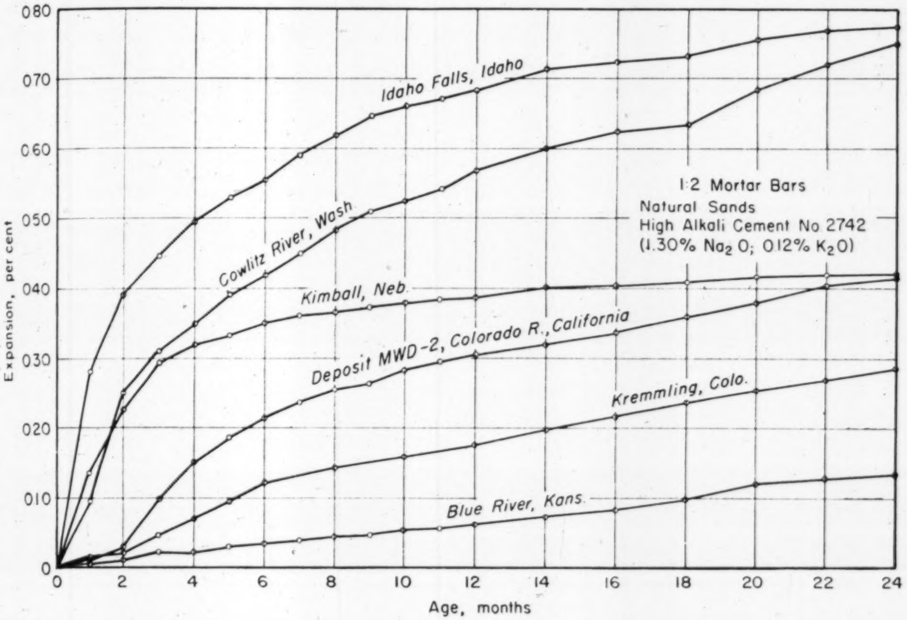


Fig. 3.—Typical Expansion of Moist Stored Mortars Containing Natural Aggregates and High-alkali Cement.

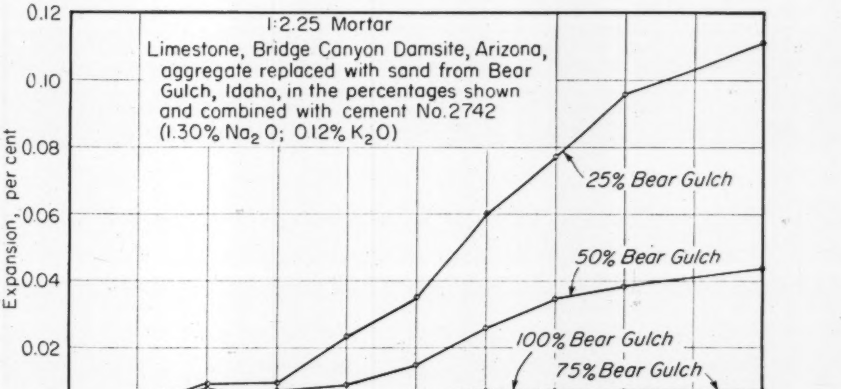


Fig. 4.—Relation of Expansion of Moist-stored Mortar Containing a High-alkali Cement and Various Proportions of a Very Reactive, Natural Aggregate.

Expansion measured on mortar bars containing natural aggregate in this series range from virtually nil to approximately 0.7 per cent linearly in 1 yr. The expansion may occur uniformly, particularly if it is of small magnitude, but typically the rate of expansion declines with passing time (Fig. 3). In this mortar test, as in others, the magnitude of the expansion is affected by all characteristics of the mortar, such as air content and cement content, and by the properties of the aggregate, such as amount and identity of the reactive constituents and porosity of the particles. For these reasons the mortar test, as outlined in this paper, may not in all circumstances yield unambiguous data with respect to alkali reactivity of aggregates.

TABLE III.—RELATION OF EXPANSION OF MOIST-STORED MORTAR BARS TO ALKALI CONTENT OF THE CEMENT.

Aggregate	Cement (Lab No.)	Alkali Content of Cement, per cent			Mortar Expansion per cent[a]	
		Na_2O	K_2O	Soda equivalents[b]	6 months	1 yr.
1. Sand Bear Gulch, Idaho (25 per cent replacement of neutral quartz)	2742	1.30	0.12	1.38	0.061	0.137
	3562	1.20	0.04	1.23	0.084	0.207
	2473	0.60	0.46	0.90	0.011	0.022
	2480	0.52	0.26	0.69	0.009	0.014
	2555	0.30	0.38	0.55	0.001	0.005
	2341	0.16	0.30	0.36	0.002	0.002
2. Sand Boysen Dam, Wyoming	3656	1.01	0.40	1.27	0.134	...
	3562	1.20	0.04	1.23	0.104	...
	8149	0.10	0.59	0.49	0.001	...
	2265	0.13	0.44	0.42	−0.003	...
3. Sand Stewart Mountain Dam, Arizona	3562	1.20	0.04	1.23	0.093	...
	2473	0.60	0.46	0.90	0.026	...
	2480	0.52	0.26	0.69	0.021	...
	2448	0.30	0.42	0.58	0.005	...
	2555	0.30	0.38	0.55	0.017	...
	7677	0.24	0.44	0.53	0.004	...
	7659	0.19	0.45	0.49	0.018	...
	7658	0.31	0.25	0.47	0.007	...
	2265	0.13	0.44	0.42	0.005	...
	3293	0.10	0.26	0.27	−0.006	...
	2735	0.04	0.14	0.13	0.002	...
4. Sand Green Mountain Dam, Colorado	2742	1.30	0.12	1.38	0.121	0.183
	MS	0.28	0.64	0.70	0.003	0.003
	2754	0.22	0.67	0.66	0.014	0.025
	4500	0.13	0.50	0.46	0.003	0.002
	2735	0.04	0.14	0.13	0.000	0.001
5. Sand Gibraltar Dam, California	2742	1.30	0.12	1.38	0.170	0.306
	3619	0.81	0.44	1.10	0.162	0.392
	2555	0.30	0.38	0.55	−0.004	0.006
	2735	0.04	0.14	0.13	−0.024	−0.012
6. Sand Kimball, Nebraska	FP	1.36	0.17	1.47	0.249	0.288
	2742	1.30	0.12	1.38	0.353	0.382
	3656	1.01	0.40	1.27	0.297	0.343
	1356	0.78	0.66	1.21	0.200	0.266
	SLH	0.65	0.45	0.95	0.227	0.334
	2473	0.60	0.46	0.90	0.162	0.300
	2480	0.52	0.26	0.69	0.121	0.326
	FC	0.24	0.60	0.63	0.079	0.243
	1782	0.58	0.08	0.63	0.026	0.145
	1592	0.40	0.29	0.59	0.025	0.119
	2555	0.30	0.38	0.55	0.064	0.179
	4043	0.40	0.14	0.49	0.014	0.070
	2265	0.13	0.44	0.42	0.010	0.013
	2735	0.04	0.14	0.13	0.004	0.004

[a] Mortar bar procedure as indicated in Appendix III, except that for Aggregates Nos. 1, 2, and 3, mix parts are 1:2.25 and for aggregates Nos. 2 and 3, gradings are those used in construction.

[b] Weight per cent of Na_2O plus 0.658 times weight per cent of K_2O.

For example, highly reactive aggregates may cause a smaller expansion than considerably less reactive aggregates because the content of reactive constituents exceeds the "pessimum" proportion. Yet, because of possible variations in composition of the aggregate from place to place in the deposit, it is dubious wisdom to permit use of highly reactive aggregates with high-alkali cements even though they cause only small mortar expansion. The potential reactivity of aggregate can be indicated by petrographic analysis, or by a modification of the mortar test in which an innocuous aggregate is used to replace various proportions of the tested aggregate, such as 25, 50, and 75 per cent by weight. The importance of such a replacement series is demonstrated by mortar tests on a natural sand from Bear Gulch, Idaho, investigated as a source of aggregate for repairs to the spillway of Grassy Lake Dam (Fig. 4). When this sand constitutes the entirety of the aggregate, mortar expansion is negligible; but when this sand constitutes only 25 or 50 per cent by weight of the aggregate (the remainder being a nonreactive, crushed limestone), significant expansion occurs. Consequently, those laboratories to which the services of an experienced petrographer are not available should enlarge upon the described procedure by introducing mortar specimens in which the tested aggregate is in part replaced by an aggregate known to be innocuous.

In Bureau of Reclamation practice, aggregates finally selected for use in major construction are tested in the mortar-bar procedure with cements of intermediate alkali content, especially in the range 0.4 to 0.7 per cent soda equivalents,[6] including, if possible, cements which may ultimately be used at the project. In addition, where considered necessary, mortars are prepared with the selected aggregates, using the proposed field grading and mix parts to simulate the characteristics of the mortar to be included in the field concrete. In this

TABLE IV.—COMPARISON OF EXPANSION OF CONCRETE TO EXPANSION OF MORTAR CONTAINING HIGH-ALKALI CEMENT.

Source of Aggregate		Concrete[a] Expansion, per cent				Mortar[b] Expansion, per cent		
		1 yr.	2 yr.	4 yr.	6 yr.	6 months	1 yr.	2 yr.
1. Coolidge Dam, Arizona	Sand Gravel	0.001	−0.001	−0.002	−0.004	0.301 0.262	0.459 0.360	0.592 0.414
2. Pasco, Washington	Sand Gravel	0.004	−0.002	−0.003	...	0.026 ...	0.093 ...	0.144 ...
3. Owyhee Dam, Oregon	Sand Gravel	0.003	0.001	0.001	...	0.544 0.360	0.656 0.432	0.715 0.491
4. Gibraltar Dam, California	Sand Gravel	−0.034	−0.043	...	...	0.170 ...	0.306 ...	0.388 ...
5. Kimball, Nebraska	Sand Sand Gravel	0.150	0.224	0.246	0.247	0.353 0.134[c] 0.192[c]	0.382 0.134[c] 0.200[c]	0.420 ... ...
6. Sand, Oro Fino Creek California; crushed quartz coarse aggregate	Sand Quartz	0.239	0.340	0.374	0.376	0.390 0.029	0.429 0.044	0.460 0.088

[a] Concrete bars are 3 by 3 by 15 in. in size, mix parts 1:2.72:2.72 (except Aggregate No. 2 with mix parts 1:2.62:3.13); W/C ranging from 0.52 to 0.57; sand graded 19 per cent by weight each Nos. 8, 16, 30, 50, 100; 5 per cent pan; gravel graded 50 per cent each No. 4 – $\frac{3}{8}$ in., $\frac{3}{8}$ – $\frac{3}{4}$ in.; cured 7 days in fog at 70 F., then stored at 70 F., in sealed cabinets in the presence of water; Cement No. 2742 (1.30 per cent Na_2O and 0.12 per cent K_2O) except Aggregate No. 6 in which MP cement (0.78 per cent Na_2O and 0.66 per cent K_2O) was used.

[b] Except as noted, mortar specimens fabricated, cured, and stored as indicated in Appendix III; Cement No. 2742 (1.30 per cent Na_2O and 0.12 per cent K_2O).

[c] 1 by 1 by 10 in. mortar bars; mix parts 1:3; graded 12 per cent No. 8, 20 per cent No. 16, 24 per cent No. 30, 24 per cent No. 50, 20 per cent No. 100; cured 7 days in fog at 70 F., then stored at 100 F. in sealed containers in the presence of water; MP cement (0.78 per cent Na_2O, 0.66 per cent K_2O).

---

[6] Alkali content of cement is computed as soda equivalents as the weight per cent of Na_2O plus 0.658 times the weight per cent of K_2O.

way, for each aggregate, is determined the degree to which alkali-aggregate reaction is controlled by restriction of alkali content of the cement. If the aggregate is so reactive that inordinate expansion of concrete can be expected with cement available to the project, use of measures in addition to restriction of alkali content of the cement will be necessary. The mortar expansion is decreased greatly by reduction of the alkali content to below 0.6 per cent (expressed as soda equivalents); and throughout the history of this test, when applied by the Bureau of Reclamation, significant expansion has never been observed on a moist-stored mortar containing a cement with less than 0.3 per cent alkalies (Table III).[7]

Although studies of concrete specimens have contributed greatly to the knowledge of the basic causes and controls of the alkali-aggregate reaction, attempts to develop a procedure to determine potential reactivity of aggregates by means of moist-stored concrete instead of mortar bars have been largely unsuccessful. For example, sand and gravel used at Coolidge Dam, Ariz.; Owyhee Dam, Ore., and Gibraltar Dam, Calif., all of which show evidence of deleteriousness in the structures and caused high mortar expansion, produced insignificant expansion or minor contraction in moist-stored concrete specimens to an age of several years (Table IV). On the other hand, some reactive aggregates cause large expansion of either moist-stored concrete or mortar.

The mortar bar test outlined herein has formed the most reliable and quantitative means of evaluating alkali reactivity of aggregates in the Bureau of Reclamation testing program. Unfortunately, the several months frequently required to obtain definitive results limit applicability of the method for specification testing.

WETTING AND DRYING OF CONCRETE

In 1941, as a part of the investigations of alkali-aggregate reaction in concrete, several concrete slabs were prepared and stored out-of-doors. Data available after more than 6 yr. of exposure clearly demonstrated that the wetting and drying and heating and cooling incident to natural exposure induce expansive disintegration with some aggregates considerably in excess of that occurring in moist-stored companion specimens.

In this program four slabs, 2 ft. square and 4 in. thick, were cast to investigate further the reactivity of the siliceous magnesian limestone (siliceous dolomite) from Paso Robles, Calif., originally tested by Stanton (2). Two of the slabs contain a crushed quartz aggregate, one being mixed with a high-alkali cement (No. 2742, 1.30 per cent Na_2O and 0.12 per cent K_2O), and the other with a very low-alkali cement (No. 2735, 0.04 per cent Na_2O and 0.14 per cent K_2O). The other two slabs contain an aggregate composed of crushed quartz (80 per cent by weight) and crushed siliceous magnesian limestone (20 per cent); the same cements were used. After 6½ yr. of exposure, only the slab containing the siliceous magnesian limestone and high-alkali cement shows visible evidences of distress. The pattern cracking (Fig. 5) penetrates the full thickness of the slab. Companion 3 by 3 by 15 in. beams cast from the same mix and stored in sealed containers, confirm the results obtained with the slabs, the beams containing the siliceous magnesian limestone and high-alkali cement having expanded 0.73 per cent linearly during a 6-yr. period, and the others having developed slight shrinkage. No measurements of expansion were made on the slabs.

---

[7] The only exception to this statement is the inordinate expansion of mortars containing the mineral alunite ($K_2Al_6(OH)_{12}(SO_4)_4$), which caused a kind of sulfate attack upon the cement, with production of large quantities of secondary calcium sulfoaluminate.

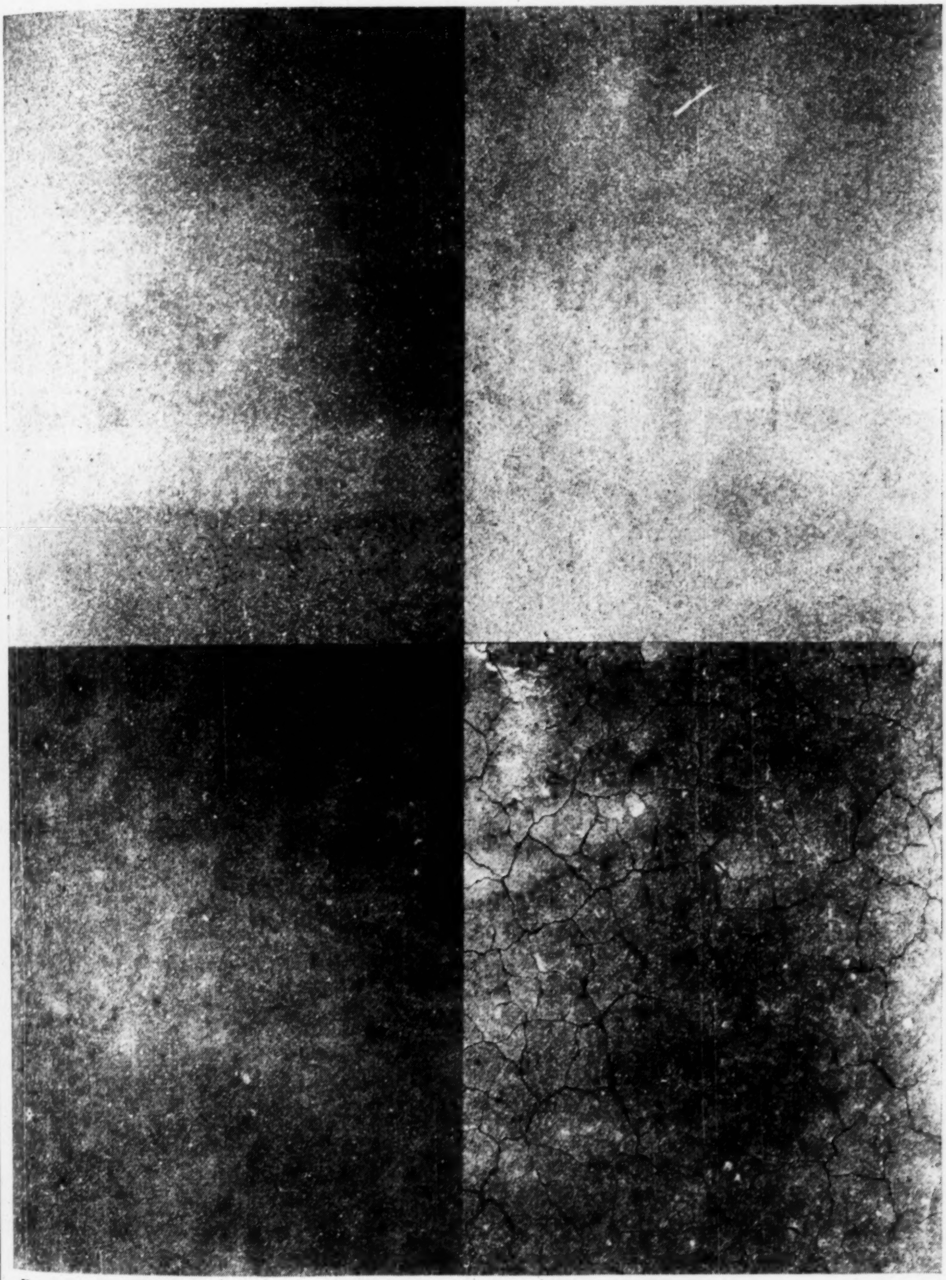


FIG. 5.—Concrete Slabs Subjected to 6½ Yr. of Natural Exposure, Demonstrating the Deterioration to be Expected When High-alkali Cements are Combined with Reactive Aggregate; Quartz Aggregate with Low-alkali (Upper Left) and High-alkali (Lower Left) Cement; Quartz with Siliceous Magnesium Limestone Aggregate and Low-alkali (Upper Right) and High-alkali (Lower Right) Cement.

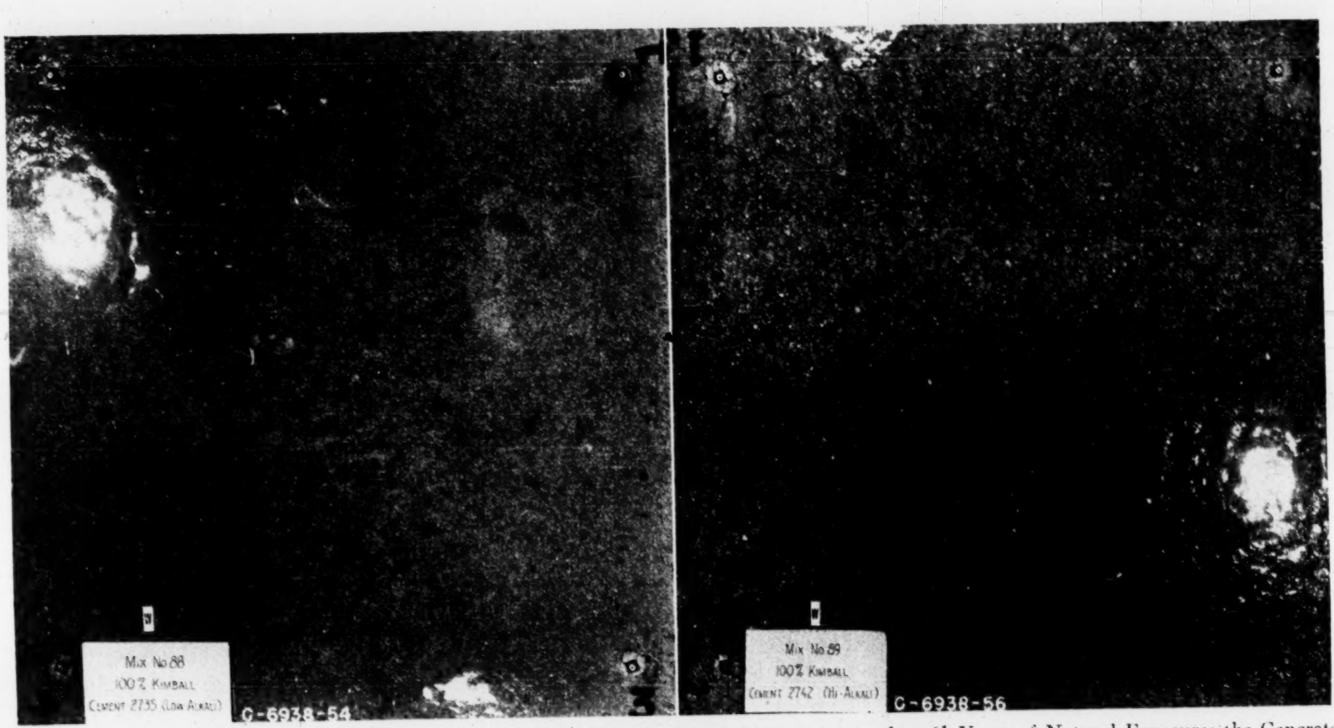

Mix No 88
100% Kimball
Cement 2735 (Low Alkali)
C-6938-54
Mix No 89
100% Kimball
Cement 2742 (Hi-Alkali)
C-6938-56

FIG. 6.—Concrete Slabs with Imbedded Reference Points for Length Change Measurement, after 6½ Years of Natural Exposure; the Concrete Contains Sand and Gravel from Near Kimball, Nebr., and Low-alkali (Left) and High-alkali (Right) Cements.

Similar slabs with reference points embedded at the corners were cast with the sand and gravel used in pavement at Kimball, Nebr., and the high- and low-alkali cements (Nos. 2742 and 2735). Street and sidewalk pavements in Kimball showed severe expansion and distress in less than 1 yr. after construction where high-alkali cement was used. Similarly, after only several months of out-of-door exposure, the slab containing the high-alkali cement showed severe expansion and pattern cracking; after $6\frac{1}{2}$ yr. of exposure, the concrete in the slab has expanded more than 0.8 per cent linearly and the cracking is pronounced (Fig. 6). Concrete beams cast from the same mix and stored in sealed containers have expanded only about one-third as much as the slab during this period. Expansion of the slab containing the low-alkali cement first was noted at an age of $5\frac{1}{4}$ yr. when the expansion was 0.05 per cent linearly. At an age of $6\frac{1}{2}$ yr. an expansion of 0.138 per cent is indicated, and a network of fine cracks and crazing is evident on the surface of the slab (Fig. 6). Companion beams in moist storage at 70 F. have shown only slight shrinkage. Consequently, the process causing expansion of the slab containing the low-alkali cement and the aggregate from Kimball appeared to be essentially independent of alkali-aggregate reaction.

Experience with these slabs clearly demonstrates the protection to concrete containing reactive aggregates gained by use of low-alkali cements. However, the data indicate also that alkali-aggregate reaction may be accelerated and that other disintegrative forces are introduced when concrete containing certain cement-aggregate combinations is subjected to repeated heating and cooling and wetting and drying. These conclusions have been amplified by abundant service records in areas of Kansas, Nebraska, northwestern Missouri, and western Iowa, where the so-called sand-

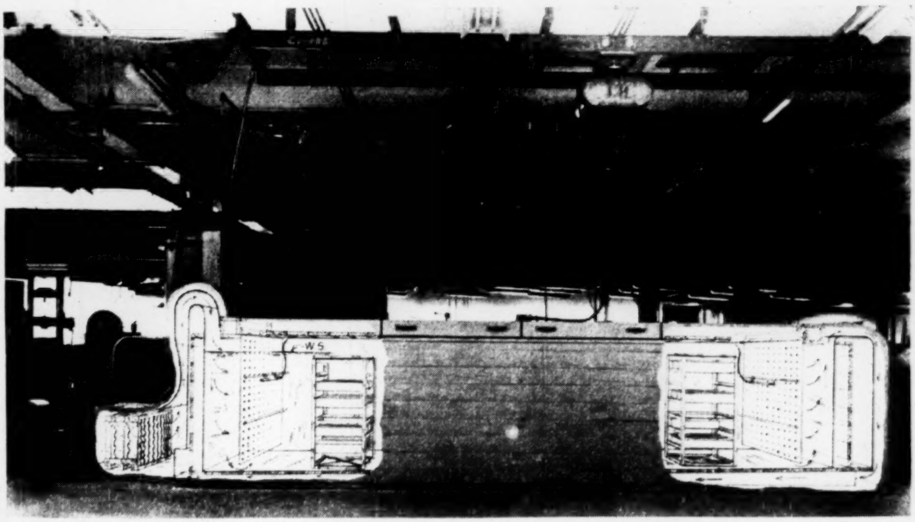


FIG. 7.—Apparatus for Automatic Operation of the Wetting and Drying Test of Concrete; capacity: 672 3 by 3 by $16\frac{1}{4}$-in. Specimens; One Cycle per Day.

gravel aggregates from deposits on the Kaw, Arkansas, Republican, Platte, and other rivers were used in concrete construction (**12**). As is true of the slab containing the aggregate from Kimball, the expansive deterioration of exposed concrete containing the sand-gravel aggregates is not prevented by limitation of alkali content of the cement (**13**).

On the basis of these observations and following the lead of C. H. Scholer (**14**), a procedure involving wetting at 70 F. and drying in air at 130 F. of 3 by 3 by 15 in. concrete beams has been incorporated in the program of tests to determine cement-aggregate incompatibility. The cycle of wetting and drying is patterned after Exposure 2 used at Kansas State College, a procedure found to induce deterioration of concrete analogous to that observed in many structures. Details of the procedure and equipment are compiled in Appendix IV.* The fully automatic apparatus

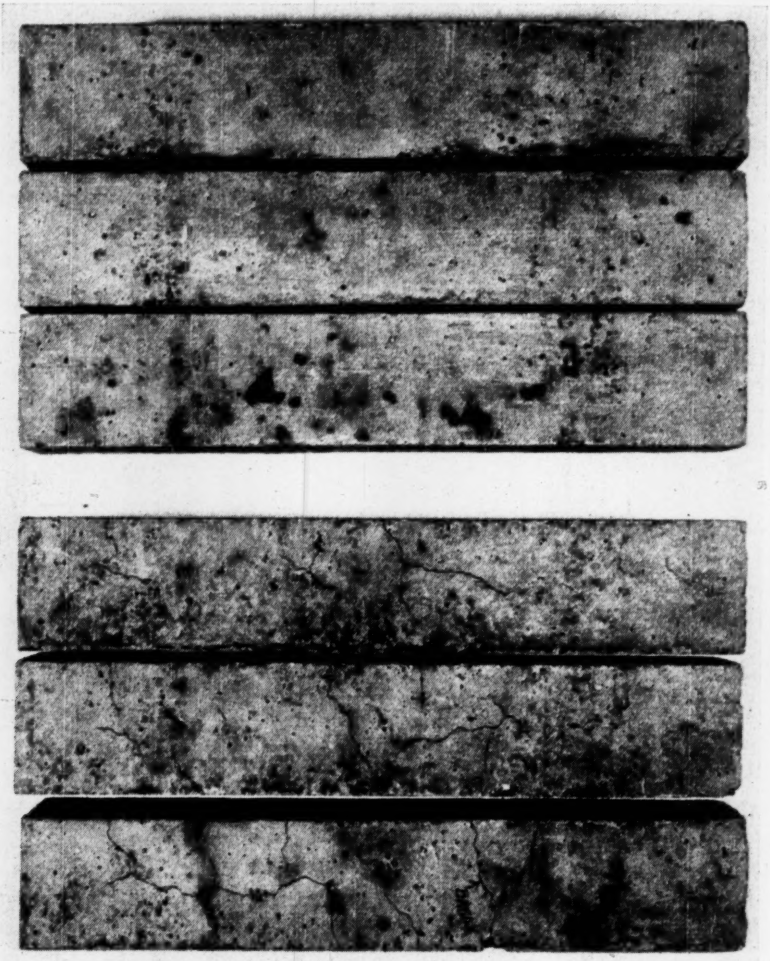


FIG. 8.—Typical Examples of Concrete Beams which have Passed (above) and Failed (below) the Wetting and Drying Test.

---

* See p. 1101.

(Fig. 7), designed and constructed by the Bureau of Reclamation, is capable of alternately wetting and drying 672 specimens; 1 cycle is obtained each day. Length change and dynamic modulus of elasticity are determined on each specimen at the end of each 28 cycles.

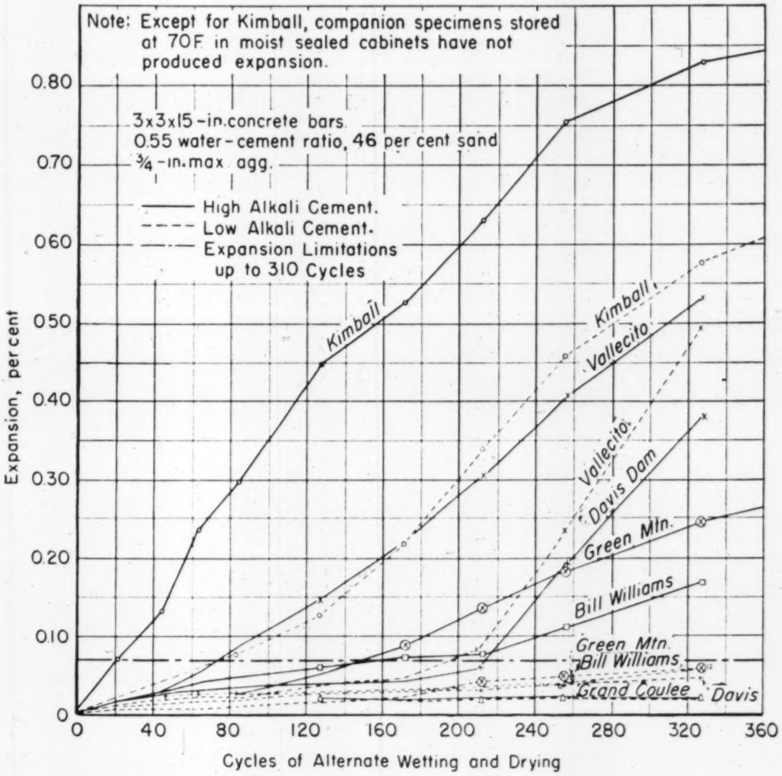


FIG. 9.—Expansion of Concrete Specimens Subjected to the Wetting and Drying Test.

During progress of the test, beams containing incompatible cement-aggregate combinations expand and crack in a manner suggestive of the effects of alkali-aggregate reaction (Fig. 8). However, lack of correlation of the progress of expansion with alkali content of the cement clearly indicates that although alkali-aggregate reaction may contribute to the expansion developed during the test, the cement-aggregate incompatibility being evaluated is essentially independent of this reaction.

For every aggreate tested, the wetting-and-drying test causes greater expansion of concrete than occurs during continuous moist storage at 70 F. Sand and gravel used in construction at Kimball, Nebr.; Vallecito Dam, Colo.; Green Mountain Dam, Colo.; Davis Dam, Ariz.-Nev.; and Parker Dam, Calif.-Ariz., all of which are aggregates known to be deleterious to concrete, develop only insignificant expansion or contraction in equivalent moist-stored concrete, yet caused expansions of concrete in excess of 0.05 per cent linearly during 310 cycles in the wetting-and-drying test (Fig. 9).

The data now indicate that expansion in excess of 0.07 per cent linearly in 310 cycles or less of this wetting-and-drying test must be regarded as failure. Addi-

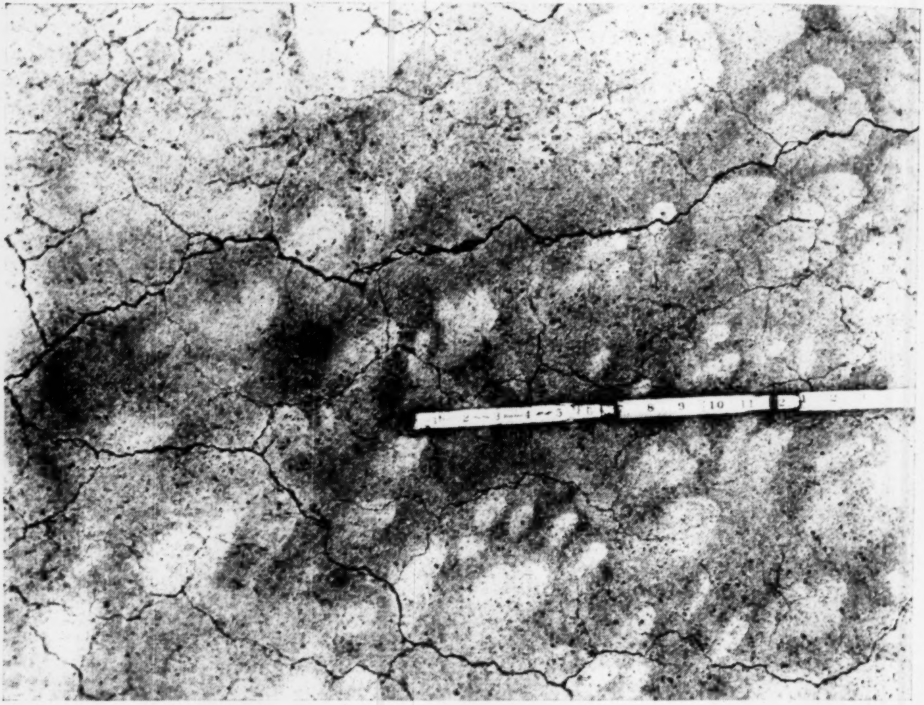


FIG. 10.—Typical Pattern Cracking on the Exposed Surface of Concrete Affected by Alkali-aggregate Reaction, Stewart Mountain Dam, Arizona.

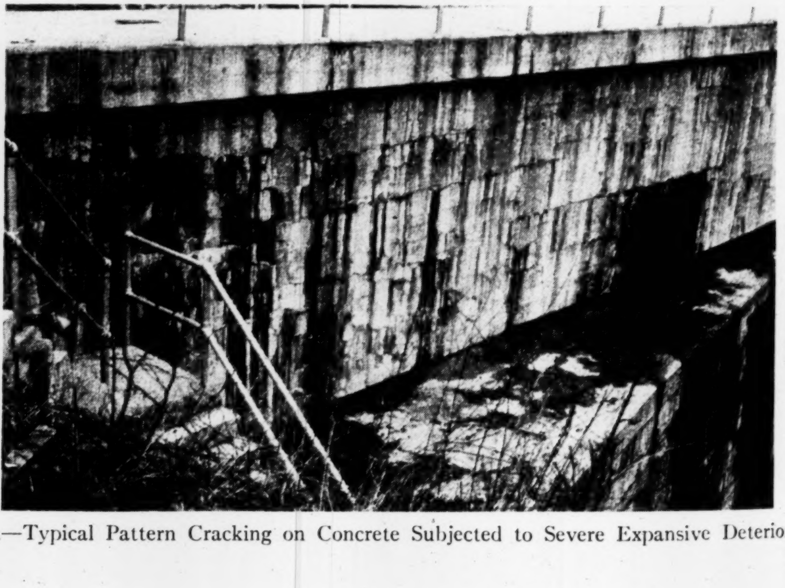


FIG. 11.—Typical Pattern Cracking on Concrete Subjected to Severe Expansive Deterioration.

tional records of field service may suggest some revision of this tentative limit.

Research to assemble a background of information to facilitate interpretation of test results and to develop means by which failure of concrete during the tests can be retarded is being carried forward.

EXAMINATION OF CONCRETE IN STRUCTURES

Where an aggregate has been used in concrete construction, critical examination of the concrete in field and laboratory may reveal the aggregate's deleterious or innocuous character. However, before reliance can be placed upon such an evaluation of alkali reactivity, considerable data must be available to establish the methods of fabrication, placement, and curing of the concrete; the composition and quality of the cement; and the conditions of service. Experience has demonstrated repeatedly that, for any of the following reasons, no ill effects may be evident in the concrete even if the aggregates are significantly alkali reactive: (1) use of low-alkali cement, or of a cement containing an admixture inhibiting alkali-aggregate reaction; (2) insufficient water to permit progress of the chemical reactions; (3) excessively porous condition of concrete; or (4) insufficient age.

Thus, an aggregate successfully used in one structure may prove to be deleterious in another, or deterioration may be confined merely to certain parts of a single structure. For example, aggregates from the Salt River, Ariz., have caused no deterioration of concrete at Mormon Flat Dam, where low-alkali cements were used, but serious distress has occurred at Stewart Mountain Dam, where aggregates from the same river were mixed with high-alkali cements. At Coolidge Dam, Ariz., extensive deterioration of concrete is restricted to those portions of the structure in which high-alkali cement was used, even though the same aggregate was used throughout.

Deterioration of concrete can take place through processes totally unrelated to alkali-aggregate reaction. Consequently, each instance of concrete failure must be investigated critically to establish the precise cause and, where alkali-aggregate reaction is in evidence, to establish the extent to which it may have been augmented by other disintegrative processes.

In most situations even experienced observers are unable to determine with certainty from field observations alone that alkali-aggregate reaction has or has not occurred in a concrete of an investigated structure. Therefore, examination of concrete in place in the structure should be supplemented by laboratory examination of carefully selected representative samples of the concrete.

The progress of alkali-aggregate reaction in concrete results in expansion and cracking, with accompanying decrease in strength, elasticity, and durability (**15**). Two kinds of fracture systems are recognizable: (1) those forming as a consequence of the irregular and variable distention of the concrete; and (2) those representing lines of structural failure where the distention of individual members is resolved into compressive, tensile, shearing, or torsional stresses of great magnitude.

Cracks relating directly to the expansion of the concrete develop the irregular pattern cracking so typical of concrete affected by alkali-aggregate reaction or other forms of deterioration accompanied by autogenous distention (Figs. 10 and 11). These cracks are widest at the surface, possibly as wide as 1.5 in. where internal expansion is very great; but they invariably extend into the concrete only for short distances, usually only a few inches, being lost in a maze of rami-

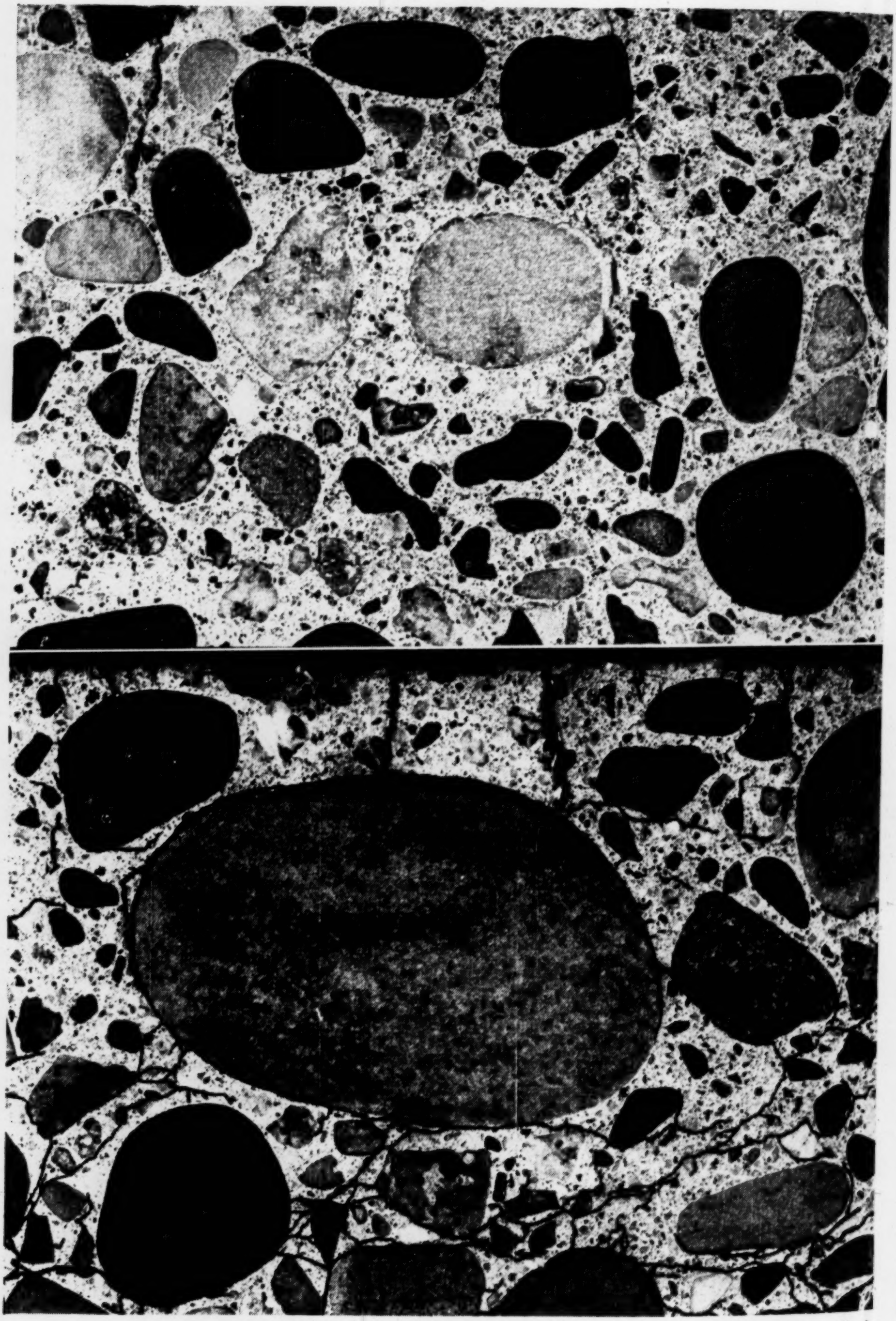


FIG. 12.—Ramifying Cracks in Concrete Seriously Affected by Alkali-aggregate Reaction; Finely Ground Surface (*Top*), Cracks Traced with Ink on Opposite Side of Section (*Bottom*). Walkway Surface at Top of Stewart Mountain Dam, Arizona.

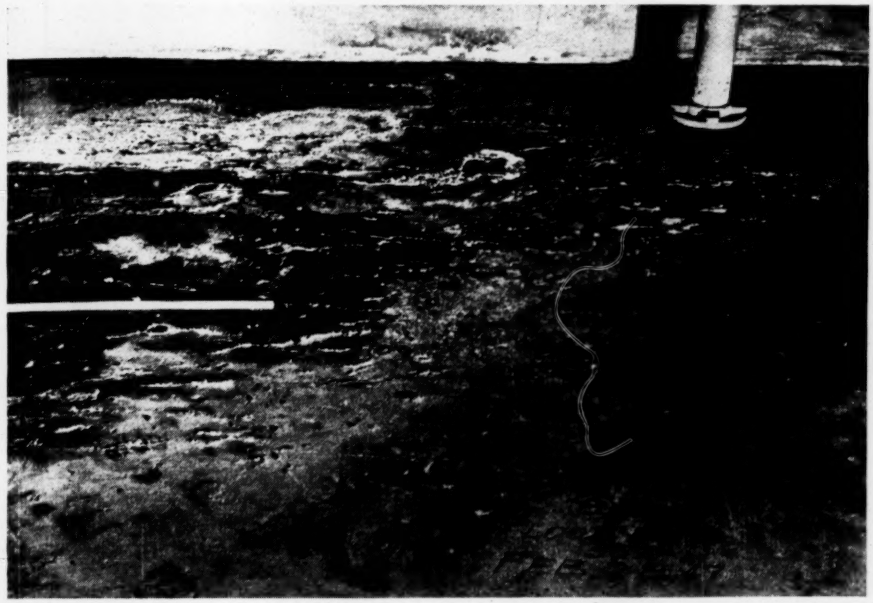


FIG. 13.—Exudations of Alkali Silica Gel and Efflorescent Deposits Formed on Concrete Floor Affected by Alkali-aggregate Reaction.

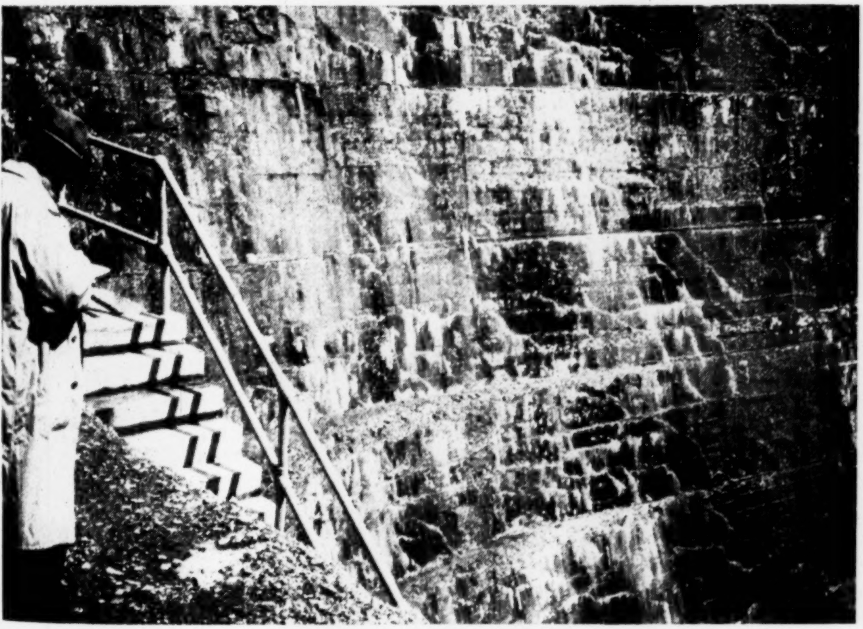


FIG. 14.—Severe Cracking and Formation of Efflorescent Deposits on Concrete Possibly Affected by Alkali-aggregate Reaction.

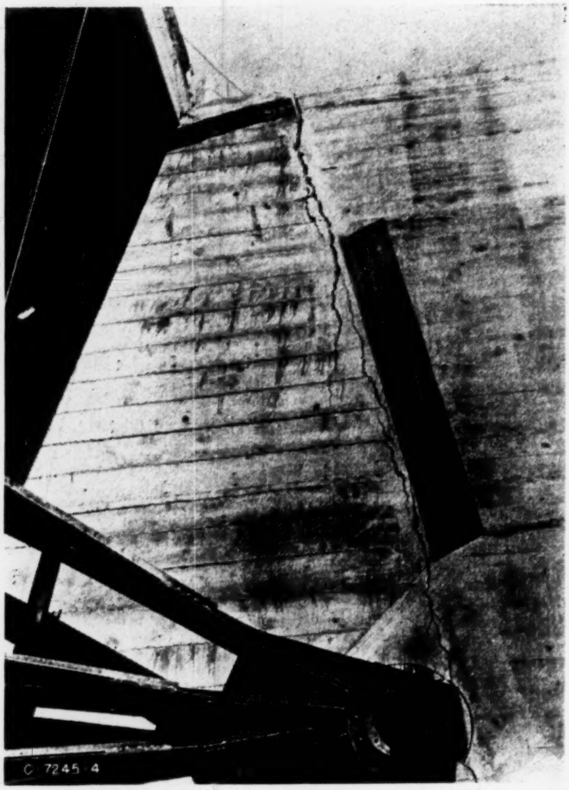


FIG. 15.—Structural Failure of Concrete, Stewart Mountain Dam, Arizona.

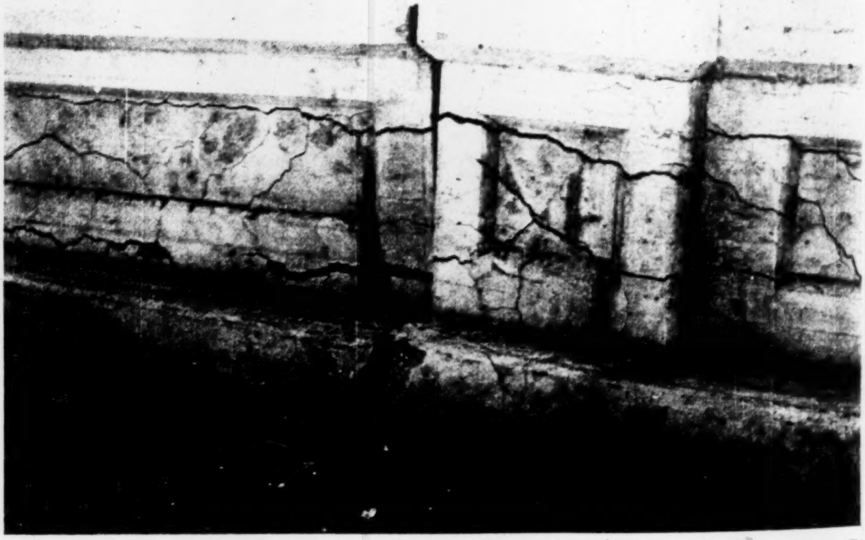


FIG. 16.—Parapet Wall Cracked and Expanded, with Resulting Dislocation near Ventura, Calif.

fying fractures of microscopic dimensions (Fig. 12). These surface cracks frequently afford avenues for seepage of water; consequently they may become the locus of extrusion of alkali silica gel and deposition of efflorescent materials leached from the concrete (Figs. 13 and 14).

Lines of structural failure demonstrate a high degree of distress in the concrete. For this reason, their development must be considered critically significant in analysis of the stability of the structure. Moreover, such cracks may seriously impair operation of appurtenant machinery (Fig. 15).

During examination of a structure, expansion of the concrete frequently can be demonstrated in several ways. Expansion joints may be partially or completely closed; relative dislocation of portions of structures may occur, causing members to shift with respect to one another (Fig. 16), shafts and gates to bind, and joints to be offset (Fig. 17). Repeated survey of points or reading of strain gages embedded in the concrete may establish quantitatively the magnitude and rate of expansion. For example, surveys at Copper Basin Dam, Calif., indicate that the crown of the dam deflected upstream at a more or less uniform rate a total of 4.8 in. in the 9 yr. following construction, that in the 6 yr. from April, 1941, to April, 1947, the concrete expanded an average of 0.015

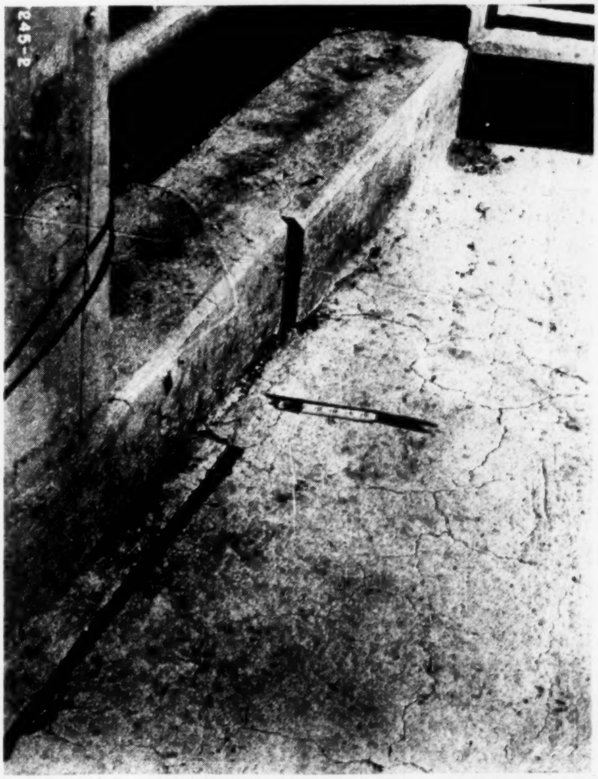


FIG. 17.—Offset Joint and Typical Pattern Cracking of Concrete Affected by Alkali-aggregate Reaction, Stewart Mountain Dam, Arizona.

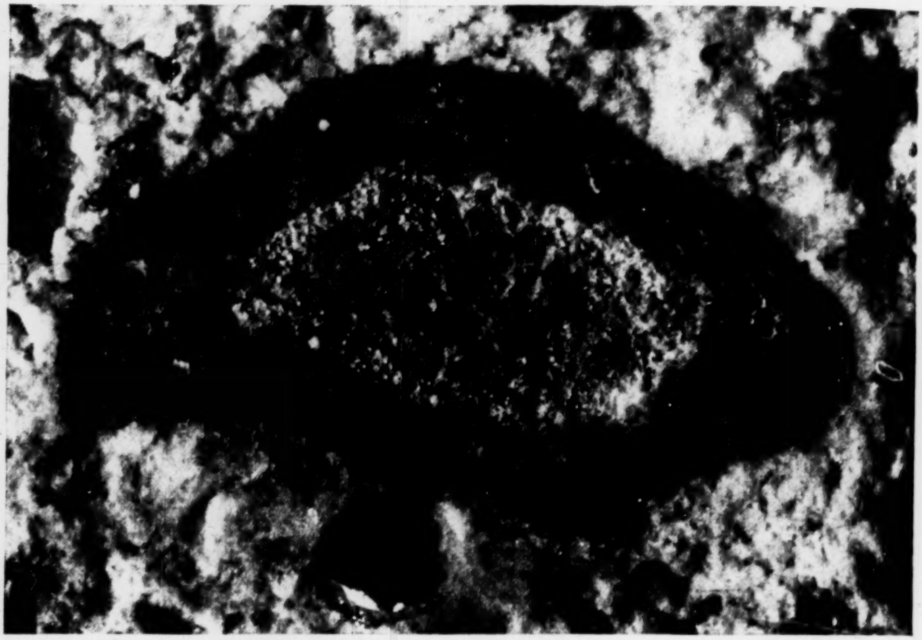


FIG. 18.—Reaction Rim on Rhyolite Grain and Associated Body of Alkali Silica Gel in Concrete from Stewart Mountain Dam, Arizona. Magnification ×8.

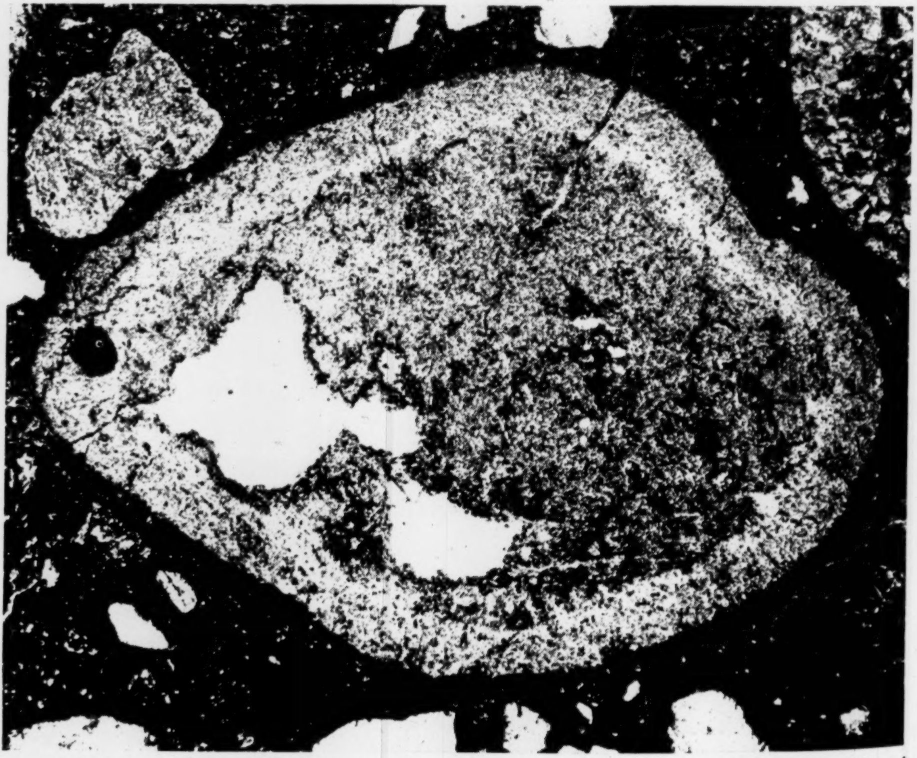


FIG. 19.—Photomicrograph Showing "Clarified" Reaction Rim on an Andesite Pebble in Affected Concrete. Magnification ×35.

per cent per year vertically, and that in the period from October, 1942, to April, 1946, the concrete expanded 0.013 per cent per year horizontally along the arc of the dam. Measurements at Parker Dam, Calif.-Ariz., demonstrate that expansion of the concrete increases from the surface to a depth of 10 ft.; and expansion in excess of 0.1 per cent linearly has been detected (**15**). Expansion of concrete in structures as a result of alkali-aggregate reaction can exceed 0.5 per cent linearly.

With detailed examination of the concrete, particularly microscopical examination in the laboratory, several phenomena diagnostic of alkali-aggregate reaction can be identified, whereby the existence of alkali-aggreate reaction can be proved even in the absence of the physical manifestations of expansion and deterioration indicated above. There

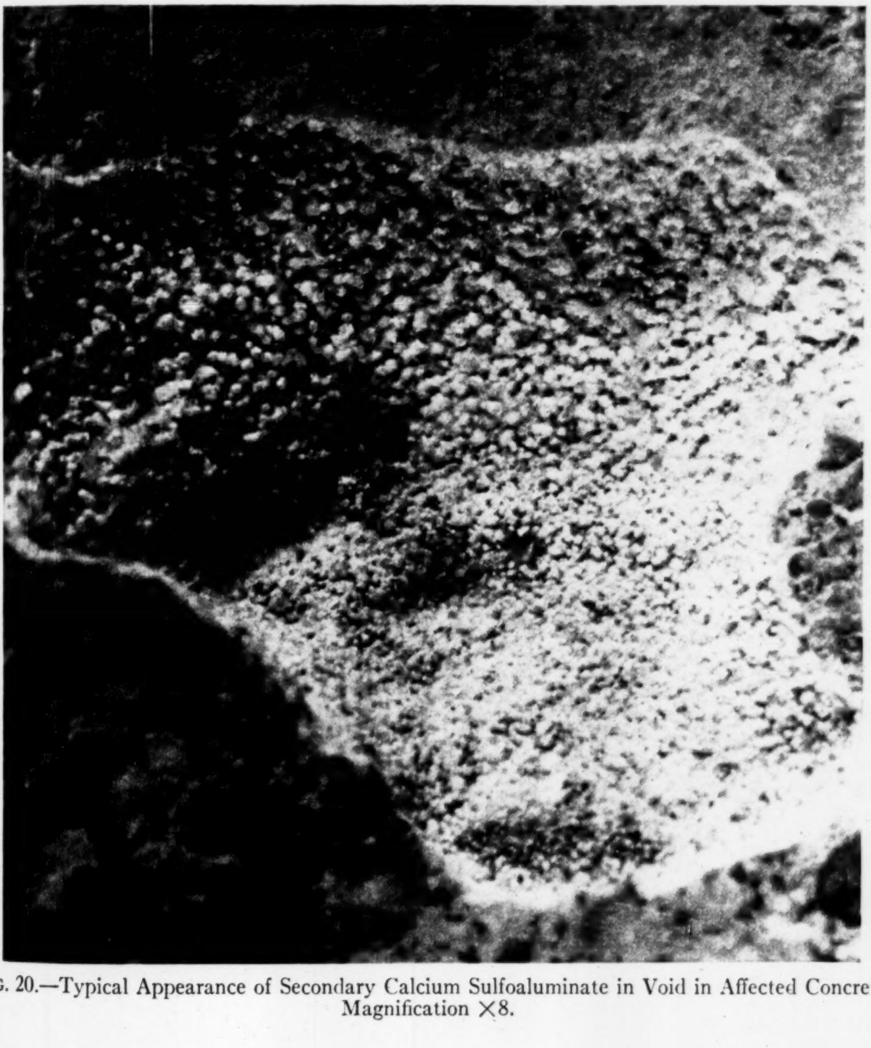


FIG. 20.—Typical Appearance of Secondary Calcium Sulfoaluminate in Void in Affected Concrete. Magnification ×8.

are two criteria which permit recognition of alkali-aggregate reaction in concrete: (1) the presence of alkalic silica gel; and (2) the occurrence of reaction rims upon particles of aggregate (16).

The alkalic silica gel is formed by interaction of the cement alkalies with the susceptible rocks and minerals of the aggregate. Consequently, these gels are present in concrete only if alkali-aggregate reaction has occurred. They accumulate within pores in the aggregate or cement paste, in fractures, and as exudations upon the surface at cracks or joints, or in association with popouts. The silica gels vary in properties because of variations in water content and degree of carbonation from watery to viscous to hard. When exposed to the atmosphere, the gels are rapidly desiccated and sometimes carbonated, becoming white and porcelaneous. At Stewart Mountain Dam, Ariz., highly fluid, clear siliceous gel still exudes from the concrete 19 yr. after completion of construction.

Reaction rims represent the peripheral zone of an aggregate particle in which dissolution and decomposition of original constituents have occurred as a result of the attack of the highly alkaline solutions permeating the concrete (Fig. 18). On fractured surface in reflected light, the rims appear dark; but when viewed microscopically in transmitted light, the rims have a higher transparency than the interior of the particle (Fig. 19). In examination of concrete to establish the existence of alkali-aggregate reaction, care must be taken to distinguish these reaction rims from weathered rims formed on the aggregate particles before the concrete was produced.

Other phenomena commonly occur in concrete affected by alkali-aggregate reaction, but not exclusively so. Thus, their presence may indicate the progress of deterioration but cannot be used alone

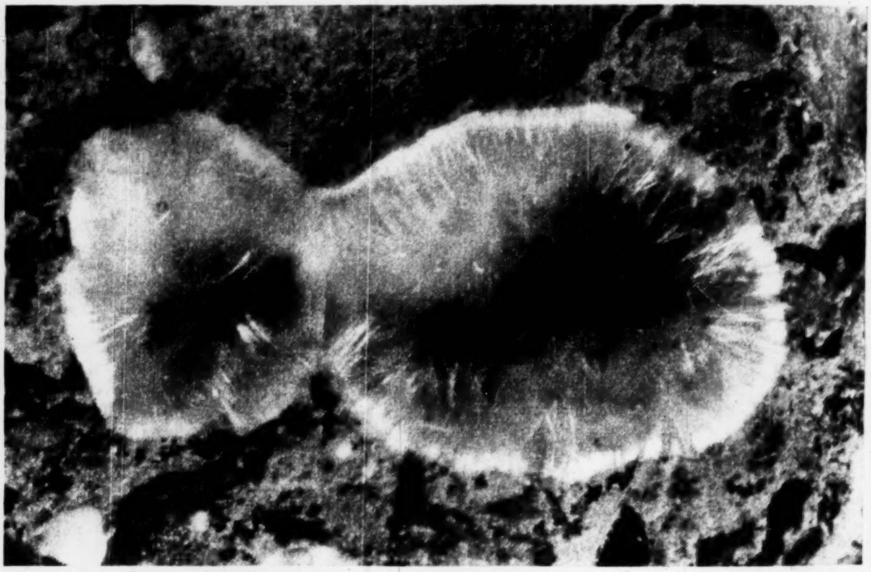


FIG. 21.—Fibrous Crystals of Secondary Calcium Sulfoaluminate in Void in Affected Concrete. Magnification ×12.

to prove that alkali-aggregate reaction has occurred. These phenomena are: (1) microfractures in the mortar; (2) deposits of fibrous calcium sulfoaluminate (Figs. 20 and 21); and (3) carbonation of the cement paste.

CONCLUSIONS

A deleterious degree of alkali-reactivity is indicated if the aggregates are shown to contain proportions of reactive substances in excess of 0.25 to 5 per cent by weight, depending upon the identity of the reactive constituents; are characterized by a ratio S_c/R_c[8] greater than 1 as a result of the chemical test; cause expansion of the moist-stored mortar specimens in excess of 0.1 per cent linearly in a year with the high-alkali cement; cause expansion of the concrete beams in excess of 0.07 per cent linearly in 310 cycles of wetting and drying; or are known to be associated with the characteristic expansive deterioration of concrete in structures. Prescribed test procedures must be followed to derive data correlative with the indicated limits.

The petrographic examination and chemical test can be completed within three work days, and they do not require elaborate equipment. The tests of moist-stored mortar and of concrete subjected to repeated wetting and drying frequently do not yield definitive results for several months; they are expensive and require extensive facilities. Consequently, in the Bureau of Reclamation procedure the petrographic examination and chemical test only are applied in investigations directed to selection of aggregates. The tests of mortar and concrete are used primarily to corroborate the indicated innocuous or deleterious nature of the aggregates which have been selected for use and to ascertain the degree to which expansion due to alkali-aggregate reaction is reduced by use of cements of reduced alkali content or by other means. These procedures are supplemented by service records if the tested aggregate has been used previously in concrete construction.

Correlation of the results of the described tests with service histories of concretes indicates that the procedures can be applied with confidence wherever alkali reactivity of aggregates is suspected.

REFERENCES

(1) T. E. Stanton, "Influence of Cement and Aggregate on Concrete Expansion," *Engineering News-Record*, Vol. 124, p. 171 (1940).

(2) T. E. Stanton, "Expansion of Concrete Through Reaction Between Cement and Aggregate," *Proceedings*, Am. Soc. Civil Engrs., Vol. 66, p. 1781 (1940).

(3) H. S. Meissner, "Cracking in Concrete Due to Expansive Reaction Between Aggregate and High-Alkali Cement as Evidenced in Parker Dam," *Proceedings*, Am. Concrete Inst., Vol 37, p. 549 (1941).

(4) R. C. Mielenz, "Petrographic Examination of Concrete Aggregates," *Bulletin*, Geol. Soc. Am., Vol. 57, p. 309 (1946).

(5) Roger Rhoades and R. C. Mielenz, "Petrography of Concrete Aggregate," *Proceedings*, Am. Concrete Inst., Vol. 42, p. 581 (1946).

(6) F. B. Hornibrook, H. Insley, and L. Schuman, "Notes on the 'Effect of Alkalies in Portland Cement on the Durability of Concrete'," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 218 (1943).

(7) A. R. Alderman, Principal Research Officer, Division of Industrial Chemistry, Council for Scientific and Industrial Research, Australia. Personal communication.

(8) R. C. Mielenz, K. T. Greene, and E. J. Benton, "Chemical Test for Reactivity of Aggregates with Cement Alkalies; Chemical Processes in Cement-aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol. 44, p. 193 (1948).

---

[8] For definition of S_c and R_c see p. 1074.

(9) T. E. Stanton, Author's closure following Reference (**2**), *Transactions*, Am. Soc. Civil Engrs., Vol. 68, Part 2, p. 85 (1942).

(10) Bureau of Reclamation, *Proceedings of Conferences for Discussion of Problems Related to Alkalies in Cement and Their Effect on Aggregates and Concretes*, (Mimeographed) Bureau of Reclamation, Denver, Colo. (1941).

(11) R. F. Blanks and H. S. Meissner, "The Expansion Test as a Measure of Alkali-aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol. 42, p. 517 (1946).

(12) W. E. Gibson, "Use of Sand-gravel Aggregate in Concrete," *Proceedings of the Kansas Highway Engineering Conference, Kansas State College Bulletin*, Vol. 30, p. 83 (1946).

(13) A. D. Conrow, Discussion of a paper by W. C. Hanna, "Unfavorable Chemical Reactions of Aggregates in Concrete and a Suggested Corrective," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 1000 (1947).

(14) Engineering Experiment Station of Kansas State College and the Portland Cement Association, *A Study of Special Concrete Problems in Kansas, Nebraska, Iowa, and Western Missouri*, Part I (1946).

(15) R. F. Blanks and H. S. Meissner, "Deterioration of Concrete Dams Due to Alkali-aggregate Reaction," *Transactions*, Am. Soc. Civil Engrs., Vol. 111, p. 743 (1946).

(16) Duncan McConnell, R. C. Mielenz, W. Y. Holland, and K. T. Greene, "Cement-Aggregate Reaction in Concrete," *Proceedings*, Am. Concrete Inst., Vol. 44, p. 93 (1948).

APPENDIX I

PETROGRAPHIC EXAMINATION OF CONCRETE AGGREGATE TO DETERMINE CHEMICAL REACTIVITY WITH CEMENT ALKALIES

Screen sufficient aggregate to provide at least the following quantities of the various sizes:

Size of Aggregate	Weight, g.
1½ to ¾ in.	4000
¾ to ⅜ in.	1000
⅜ to 3/16 in.	200
3/16 in. to No. 8	100
No. 8 to No. 16	50
No. 16 to No. 30	25
No. 30 to No. 50	25
No. 50 to No. 100	25
Minus No. 100 pan	10

The examination of the aggregate is performed by a qualified petrographer who is familiar with problems of concrete. Each of the size fractions is examined individually.

The samples are examined by the petrographer to determine the extent to which the particles are coated with silt, clay, or other mineral substances. The composition of the coating is determined by microscopical, chemical, or other techniques, as required. The samples are washed to remove coatings of silt and clay on the aggregate particles, and then dried.

The samples of coarse aggregate are examined particle by particle, each particle being broken on the anvil by the blow of a mallet and identified by means of the hand lens, if adequate, or microscopically. The individual particles are segregated according to three criteria: (1) Petrographic identity, (2) physical condition, and (3) anticipated chemical stability in concrete. Each category shall be classified according to a rock name (for example, granite or deeply weathered basalt) and a designation of chemical reactivity according to the following scale:

> "Deleterious" materials produce adverse effects on concrete due to chemical reaction which takes subsequent to hardening of the cement. Substances designated as "deleterious" by reason of susceptibility to attack by alkalies in cement are not deleterious if used with a cement of low alkali content.
>
> "Innocuous" constituents do not participate in chemical reactions harmful to concrete.

Ordinarily, complete segregation of fine aggregate fractions is not justified, but if present, physically unsound or chemically deleterious particles are segregated and identified petrographically, and appropriate description of their properties made. For all aggregates the fraction passing the No. 50 screen is examined in immersion oils under the petrographic microscope to determine the presence or absence and the approximate abundance of deleterious substances.

Following the completion of his examination and analysis, the petrographer prepares a report summarizing his observations and conclusions regarding the suitability of the aggregate under the anticipated conditions of service, indicating any necessary qualifications to its use or the need for special tests to elucidate the significance of particular properties.

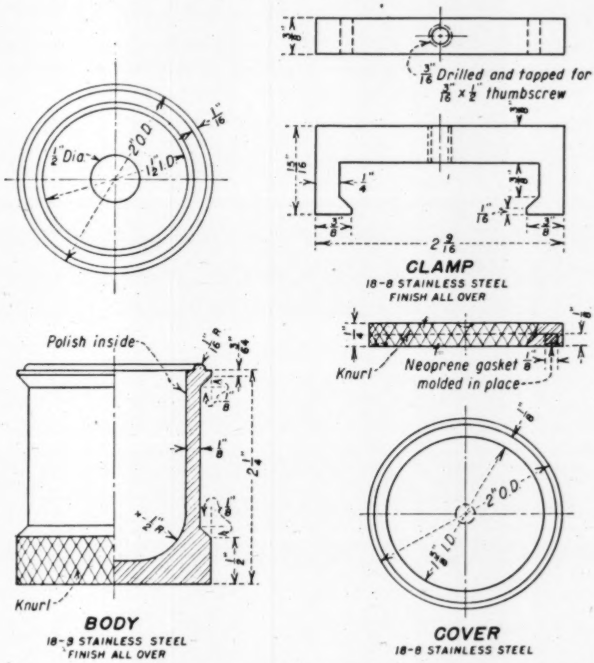
APPENDIX II

CHEMICAL TEST TO DETERMINE REACTIVITY OF CONCRETE AGGREGATE WITH CEMENT ALKALIES

Procedure:

Screen sufficient aggregate to provide at least the following quantities of the various size fractions: 1000 g. each of the $1\frac{1}{2}$ to $\frac{3}{4}$ in., $\frac{3}{4}$ to $\frac{3}{8}$ in., and $\frac{3}{8}$ to $\frac{3}{16}$ in.; and 200 g. each of the $\frac{3}{16}$ in. to No. 8, No. 8 to No. 16, No. 16 to No. 30, No. 30 to No. 50, and No. 50 to No. 100 sizes. Prepare a sample of coarse aggregate to be tested by combining 1000 g. of each of the three indicated coarse aggregate sizes, then crush and repeatedly screen the composite sample over the No. 50 and No. 100 screens until virtually all of the material passes the No. 50 screen. Wash the material retained on the No. 100 screen to remove the dust and undersize material. Prepare similarly the sample of fine aggregate for use in the chemical test, except that the composite may be prepared by combination of 200 g. each of the five sand sizes. Dry the sample to constant weight at 110 C.

Weigh out in duplicate 25.0 g. of the dry No. 50 to No. 100 material into small stainless steel or other nonreactive metal containers (Fig. 22). Add 25 ml. of freshly prepared 1.000 ± 0.010 N NaOH solution. Stir the mixture to remove entrapped air; then seal the containers. Immediately thereafter, place the containers in a liquid bath or oven capable of maintaining a temperature of 80 ± 1 C. for 24 hr. After 24 hr., remove the containers from the oven or bath, cool for 15 min. in water maintained at 25 C., and as quickly as possible filter the solution from the aggregate material by the following procedure. Use a porcelain Gooch crucible (Coors Size No. 4) with a disk of filter paper (equivalent to Schleicher & Schuell Co. No. 604) cut to fit the bottom of the crucible, setting the crucible in a filter tube fitted with Gooch tubing. With aspirator in operation or vacuum line open decant a small quantity of the solution onto



3/16" Drilled and tapped for 3/16" x 1/2" thumbscrew
1/2" Dia.
2" O.D.
1 1/2" I.D.
1/16"
1/4"
1 5/16"
2 9/16
3/8"
CLAMP
18-8 STAINLESS STEEL
FINISH ALL OVER
Polish inside
1/16" R
1/8"
1/2" R
2 1/4"
1/2"
Knurl
BODY
18-8 STAINLESS STEEL
FINISH ALL OVER
Knurl
Neoprene gasket
molded in place
1/8"
2" O.D.
1 5/8" I.D.
COVER
18-8 STAINLESS STEEL

FIG. 22.—Container Recommended for Use in Chemical Test with NaOH Solution.

the filter paper so that it will seat properly in the crucible. Then pour the remainder of the free-flowing solution into the crucible. With a steel spatula, remove as much as practicable of the aggregate sample from the container and pack into the crucible. Continue the filtration until all of the filtrate extractable by this method has been obtained. Do not flush the sample in any way to extract the residuum of solution absorbed within or adherent to the aggregate particles

Immediately following completion of filtration stir the solution to assure homogeneity, then take an aliquot of 10 ml. of the filtrate and dilute to 200 ml. in a volumetric flask. Determine the concentration of silica in the solution. Take an aliquot of 20 ml. of the diluted solution and titrate against 0.05 N HCl to the phenolphthalein end point.

Calculate the silica concentration and reduction in alkalinity of the NaOH solution filtered from the aggregate material as follows: $R_c = N\text{-}50\,v$ and $S_c = 20C$, where R_c equals reduction in alkalinity in millimoles per liter; S_c equals concentration of silica in millimoles per liter; v equals volume in milliliters of HCl solution used in the titration; C equals concentration of silica in the diluted solution, expressed in millimoles per liter; and N equals the concentration of the original NaOH solution (1000 ± 10), expressed in millimoles per liter.

APPENDIX III

MORTAR BAR TEST TO DETERMINE REACTIVITY OF AGGREGATE WITH CEMENT ALKALIES[9]

Materials:

Use a type II cement ground to a fineness of 1600 sq. cm. per gram or greater (Wagner Turbidimeter method), containing 1 per cent or more total alkalies (Na_2O and K_2O) expressed as soda equivalents, and a second cement containing less than 0.2 per cent total alkalies. In Bureau practice the cements used are No. 2742 (1.30 per cent Na_2O and 0.14 per cent K_2O) and No. 2735 (0.04 per cent Na_2O and 0.14 per cent K_2O) or their equivalents, the very low alkali cement being used to establish whether expansive actions other than alkali-aggregate reaction might occur. In addition, cements which may be used in construction might be included in the program to evaluate the significance of marginal degrees of reactivity.

For testing fine aggregate grade the aggregate 19 per cent by weight each of the No. 4 to No. 8, No. 8 to No. 16, No. 16 to No. 30, No. 30 to No. 50, and No. 50 to No. 100 sizes, with 5 per cent of material passing the No. 100 sieve. Crush coarse aggregate to fine aggregate sizes and grade the aggregates for the mortar 20 per cent by weight each of the five sizes, omitting the material passing the No. 100 screen. To evaluate the reactivity of aggregates containing abundant deleterious constituents, prepare similarly graded aggregate in which the test material is replaced by 25, 50, and 75 per cent, respectively, of an innocuous aggregate.

Preparation of Specimens:

Prepare in triplicate specimens 1 by 1 by 11¼ in. in size for each cement-aggregate combination, using a mix composed of 1 part cement to 2 parts aggregate by weight. The net water-cement ratio is maintained at 0.40 by weight. Some adjustment of this ratio is required for testing of lightweight aggregates to maintain a reasonably constant volume relation of cement to aggregate.

Ordinarily, 450 g. of cement, 900 g. of aggregate, and 180 g. of water are sufficient to fabricate the necessary specimens. Mix the ingredients in a bowl (enamelware or other nonabsorbent, noncorroding material) of about 1-gal. capacity by vigorous and continuous stirring, squeezing, and kneading with one hand protected by a rubber glove. Introduce the materials in the following manner: (1) Place the water in the bowl; (2) add the cement to the water and mix for 30 sec.; (3) add approximately one-half of the aggregate and mix for 30 sec.; and (4) add the remainder of the aggregate and mix for 1½ min.

---

[9] The procedure described here differs in some respects from a procedure recently recommended for this purpose (reference 11 in paper). The procedure herein described, except for size of specimen, is that initiated in 1941 to test available aggregates upon which service histories had been obtained; and it is this procedure upon which are based the indicated limits to determine the chemically deleterious or innocuous character of aggregates.

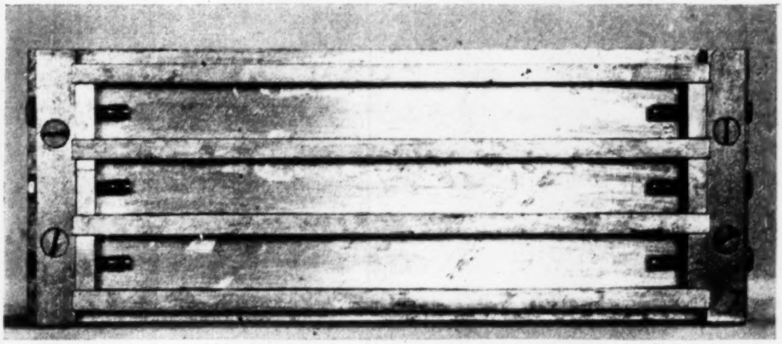


FIG. 23.—Mold for Preparation of 1 by 1 by 11¼-in. Mortar Specimens.

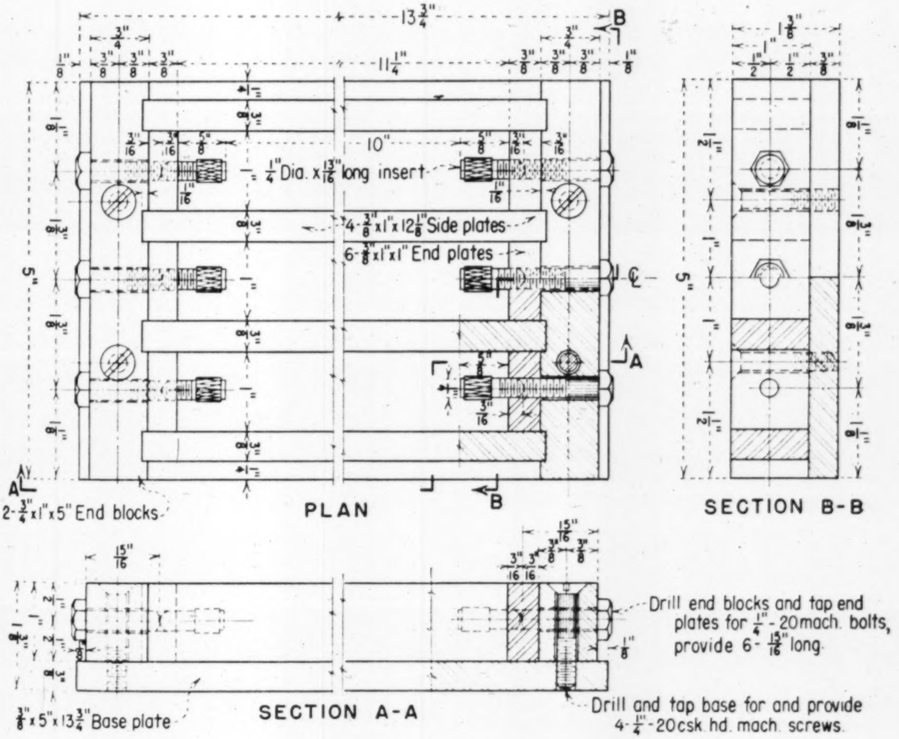

13 3/4"
3/4"
1/8"
3/8"
11 1/4"
B
10"
5/8"
3/16"
1/16"
1/4" Dia. x 13/16" long insert
4-3/8" x 1" x 12 1/8" Side plates
6-3/8" x 1" x 1" End plates
5"
1 3/8"
1"
1/2"
℄
A
2-3/4" x 1" x 5" End blocks
PLAN
SECTION B-B
15/16"
SECTION A-A
Drill end blocks and tap end plates for 1/4" - 20 mach. bolts, provide 6-15/16" long.
3/8" x 5" x 13 3/4" Base plate
Drill and tap base for and provide 4-1/4"-20 csk hd. mach. screws.

FIG. 24.—Detail of Molds Used to Fabricate 1 by 1 by 11¼-in. Mortar Specimens.

Immediately after mixing, introduce the mortar into the mold (Figs. 23 and 24) in one layer and compact the layer with the forefinger by pressing the mortar into the corners around the reference points and along the surfaces of the mold, until a homogeneous specimen has been prepared. After compaction is completed, cut off the mortar flush with the top of the mold with a straight-edged trowel, using a sawing motion, and smooth the surface with not more than two strokes with the trowel lying flat across the top of the mold. Excessive troweling will form a dense and impervious section of the specimen, which will cause warping if the aggregate is deleteriously reactive.

Curing of Specimens:

After fabrication of the specimens, immediately place the molds in a moist closet or moist room maintained at 70 F., where they are permitted to remain for 7 days. Strip molds from the specimens after 24 hr.

Storage of Specimens:

After the curing period, determine the length between outer ends of the terminal

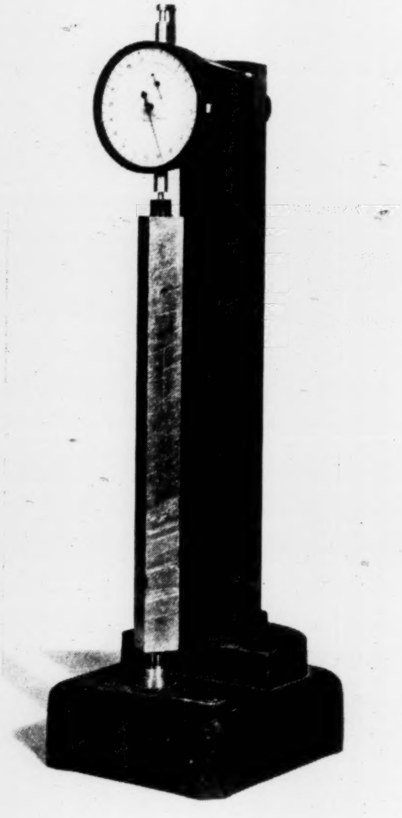


FIG. 25.—Comparator for Length Change Measurement on Mortar Specimens.

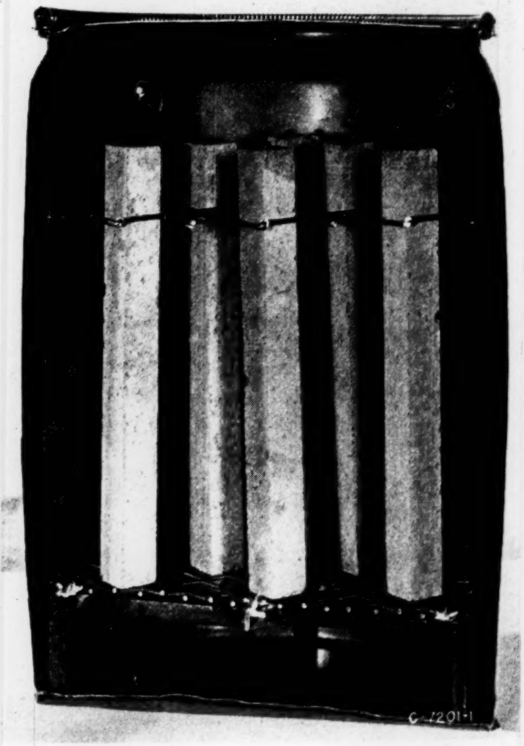


FIG. 26.—Cutaway Section of Storage Container for Moist Stored Mortar Bars. Water is Maintained in Bottom of Can below Platform.

reference points in a comparator (Fig. 25) and place the specimens vertically in a storage container in which a small amount of water has been poured to occupy a portion of the space below the platform supporting the specimens (Fig. 26). Seal the container and place it in a cabinet or room maintained at 100 F.

Periodic Measurement of Specimens:

Determine the length of specimens between outer ends of the terminal reference points at monthly intervals for 2 yr., or until required data are obtained. If desired dynamic modulus of elasticity is determined also. The specimens are examined for exudations, cracks, popouts, or warping, and if observed, notation is made on the data sheet. The containers are cooled to the temperature of the room in which readings are made (in Bureau practice 70 F.) before they are opened. Immediately after the readings have been made, the specimens are returned to the containers, and the containers sealed and stored again at 100 F.

APPENDIX IV

PROCEDURE FOR WETTING AND DRYING OF CONCRETE SPECIMENS TO DETERMINE CEMENT-AGGREGATE INCOMPATABILITY

Materials:

Use a finely ground, type II cement containing 1 per cent or more total alkalies (Na_2O and K_2O), expressed as soda-equivalents, and a second type II cement containing less than 0.2 per cent total alkalies. In Bureau practice, the cements used at present are No. 3562 (1.20 per cent Na_2O and 0.04 per cent K_2O) and No. 2735 (0.04 per cent Na_2O and 0.14 per cent K_2O). In addition, cements which may be used in construction might be included in the program to evaluate the significance of marginal degrees of reactivity.

Grade the sand as follows:

No. 4–No. 8	15 per cent by weight
No. 8–No. 16	15 per cent by weight
No. 16–No. 30	25 per cent by weight
No. 30–No. 50	24 per cent by weight
No. 50–No. 100	16 per cent by weight
Passing No. 100	5 per cent by weight

Grade the gravel as follows:

No. 4—$\frac{3}{8}$ in.	40 per cent by weight
$\frac{3}{8}$–$\frac{3}{4}$ in.	60 per cent by weight

Sand constitutes 50 per cent of the total aggregate used. Forty-four pounds of aggregate is ample for preparation of three test specimens.

Preparation of Specimens:

Prepare in triplicate specimens, 3 by 3 by 15 in. in size, of each cement-aggregate combination using a mix with a constant water-cement ratio of 0.51, the water content being allowed to vary to maintain a slump of 3 ± $\frac{1}{2}$ in. Mix parts will vary slightly with different aggregates, but usually they approximate 1 part cement to 4.6 parts aggregate by weight.

Mix the dry cement and aggregate in a $\frac{3}{4}$-cu. ft. capacity pan-type mixer for a period of 30 sec. Then add the water at 70 F. and mix the batch for 3 min., and determine the slump and unit weight.

Introduce the concrete into the molds (Fig. 27) in a single layer. First compact the concrete by rodding the mortar around the reference points and into the corners; then vibrate the molds for 20 sec. by means of a vibratory platform. After compaction is complete, cut off the concrete flush with the top of the mold with a straight-edged trowel, using a sawing motion and smooth the surface with a minimum of troweling.

Curing of Specimens:

After fabrication of the specimens, immediately place the molds in a moist room maintained at 70 F., where they are permitted to remain for 24 hr. Strip the molds and continue the moist-room curing for 7 days, after which the specimens are dried at

70 F. at 50 per cent relative humidity until they are 28 days of age. Then immerse the specimens in water at 70 F. for 48 hr.

Following this treatment, determine the weight, the length between the outer ends of the terminal reference points, and the dynamic modulus of elasticity.

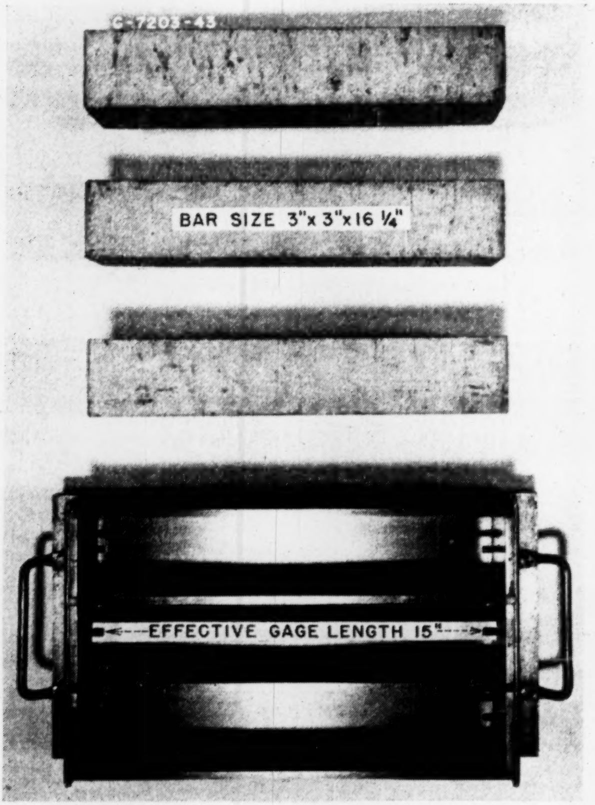


FIG. 27.—Mold and Specimens Used in the Wetting and Drying Test.

Wetting-and-Drying Treatment of Specimens:

After the initial length and dynamic modulus measurements have been obtained, dry the specimens at 130 F. in an oven or cabinet (Fig. 7)[10] for 8 hr., and then immerse in water initially at 70 F. for 16 hr. This cycle is repeated daily.

Periodic Measurement of Specimens:

Determine the increase or decrease in weight, length, and dynamic modulus of elasticity at the end of each 28 cycles (Figs. 28 and 29). Note visible features, such as cracks, exudations, popouts, or warping as they appear.

---

[10] The apparatus used by the Bureau of Reclamation comprises an insulated, steel storage tank; a three-phase, two-step, 440-v., 33-kw. heating unit; a 10½ by 15 in. centrifugal blower powered by a ½-hp. electric motor; and electrically operated 250 gal. per minute centrifugal pump; and suitable air ducts and baffles required to circulate the hot air through the cabinet. Steel racks hold and separate the test specimens, thus insuring complete coverage by water during the soaking period, and free flow of air to each specimen during the drying period.

One cycle includes 8 hr. at 130 F. with air circulating under forced draft, and changing completely each 10 to 12 sec.; and 16 hr. of soaking in water. Water enters the cabinet by gravity at a temperature of 70 F., but warms rapidly to approximately 90 F. as heat is absorbed from the tank and specimens. The temperature is reduced slowly by circulation of cool air over the water surface, until a temperature of 69 F. is reached. At this time circulation of the air is stopped. At the end of the soaking period, the water is pumped to the storage tank.

The water is re-used for a period of 30 days to minimize effects of leaching. In order to bring the air temperature to 130 F. as quickly as possible to initiate the drying period, it is necessary to recirculate 50 per cent of the heated air.

Operation of the apparatus is fully automatic.

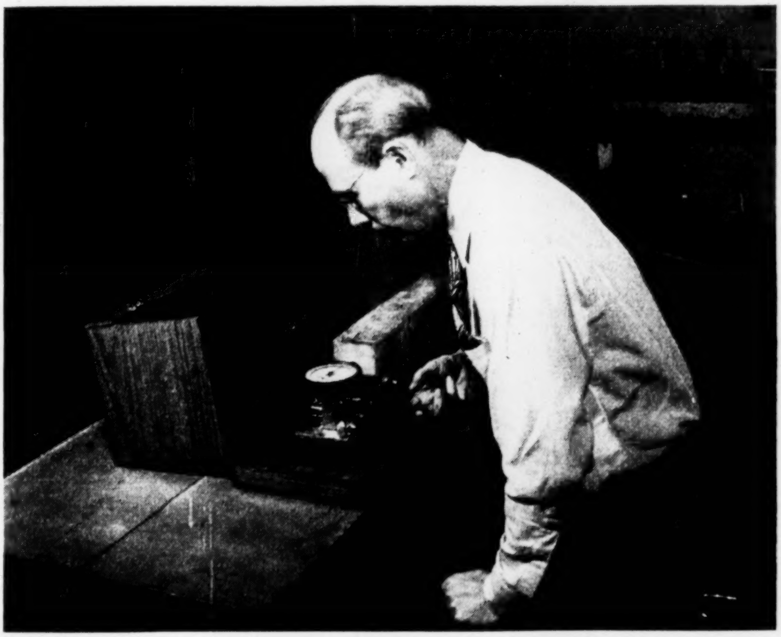


FIG. 28.—Determination of Length Change of Concrete Beams during the Wetting and Drying Test.

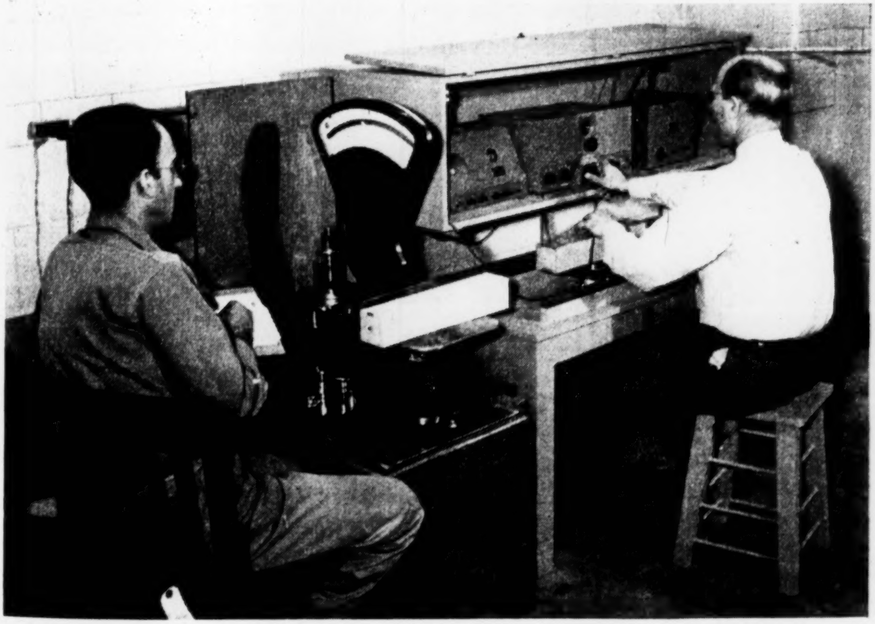


FIG. 29.—Determination of Dynamic Modulus of Elasticity and Weight of Concrete Beams During the Wetting and Drying Test.

DISCUSSION

MR. D. O. WOOLF.[1]—How is the calcium sulfoaluminate, shown in Figs. 20 and 21, identified?

MR. H. S. MEISSNER[2] (*presented paper for authors*).—I am not qualified to say. I have recognized it easily, having been shown it by the petrographers. With a lens, it appears fluffy. The crystals are acicular. They stick out like little spines, and grow as little tufts, like porcupine quills. I believe, though, it does take other forms. Perhaps Mr. Hansen could add to that.

MR. W. C. HANSEN.[3]—In the case of the calcium sulfoaluminate, the crystals are taken off and identified by the optical and other properties of the crystals.

MR. WOOLF.—Is there a chance of other chemicals being present that might be misidentified, or mistermed calcium sulfoaluminate; anything else that could possibly occur in distressed concrete which might be mistaken for calcium sulfoaluminate unless you made your optical property identification?

MR. HANSEN.—I think that would be possible. I don't know of any, but there might be.

MR. BRYANT MATHER[4].—Have the authors seen or encountered secondary gypsum in the same tufts of crystals? We frequently suspect that when we see the little groups of crystals they could conceivably be gypsum, consequently we always check them optically. To date they have always turned out to be calcium sulfoaluminate; however if gypsum were to occur it might exhibit the same appearance.

MR. W. C. HANNA[5].—I would like to refer to Table III of the paper showing the composition of the cements used. I would like to know where one would find a cement like that (1.38 per cent soda equivalent).

Other cements show 0.68 and 0.8 per cent which are to be considered high-alkali cements, getting low expansion in a year. And over in No. 4 group, you see the same thing again, a cement with 0.66, which is normally considered a high-alkali cement, the expansion is very low. As a matter of fact, the cement with 0.7 per cent alkali has only 0.003 of 1 per cent expansion which is about the same as the two that have practically no alkali.

I simply wanted to call attention to the fact that high-alkali cements are about the same as the low-alkali cements, except for the freak tests which were very high in alkali.

MR. MEISSNER.—I am very interested in Mr. Hanna's statement that he considers 0.66 and 0.68 per cent alkalies a high-alkali cement. It certainly was not considered so back in 1940. Let me assure you that every one of the cements on this chart is a commercial product, and not a freak.

Mr. Hanna asked where one would get a cement with 1.38 per cent alkalies. I will agree with him it is difficult today. I am happy that all of these investigations have had a very salutory effect on the industry. We do not find such high-alkali cements today. I am glad we do not.

---

[1] Senior Materials Engineer, Public Roads Administration, Federal Works Agency, Washington, D. C.

[2] Research Engineer, Bureau of Reclamation, U. S. Department of the Interior, Denver, Colo.

[3] Manager, Research Labs., Universal Atlas Cement Co., Buffiington, Ind.

[4] Engineer, Concrete Research Div., Waterways Experiment Station, Mississippi River Commission, Corps of Engineers, Clinton, Miss.

[5] Chief Chemist and Chemical Engineer, California Portland Cement Co., Colton, Calif.

MR. J. C. SPRAGUE.[6]—Referring to Fig. 1, I should like to ask whether there is the possibility of another curve farther to the right which might again separate innocuous from deleterious aggregate, from the standpoint of certain of those aggregates having exceeded the so-called pessimum proportions? We have tested 75 or 80 aggregates by the method developed by the Bureau of Reclamation and we have found some quite high in reduction of alkalinity and the percentage of dissolved silica which we know have not caused in a period of 15 yr. excessive expansion in the structure.

It struck me that it might add to the value of this test, which is a very short test, if there were some way to separate the innocuous from the deleterious material to the right where perhaps it crosses the pessimum line.

MR. MEISSNER.—The question is a very good one. Of course, none of these tests are infallible, and we find we must give recognition to this so-called pessimum quantity. Yes, there may be some other line to the right for various materials.

MESSRS. R. C. MIELENZ AND L. P. WITTE (*authors' closure by letter*).—As has been stated by Messrs. Hansen and Mather, calcium sulfoaluminate is identified readily by microscopical determinations and is distinguishable from other compounds occurring in concrete, such as gypsum, by its optical properties. Actually, two calcium sulfoaluminate compounds are recognized, the "high sulfate" form ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$) and the "low sulfate" form ($3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$).[7] It is the "high sulfate" form which is an almost ubiquitous ingredient of deteriorating concrete; the "low sulfate" form has not been identified in concrete, although it may possibly form during the hydration of some cements.

In our experience, the occurrence of gypsum in concrete is restricted to situations in which severe attack by sea water or sulfate-containing soils, groundwater, or wastes has taken place. However, disintegrative processes, such as freezing and thawing or alkali-aggregate reaction, without necessary accompaniment by sulfate attack, commonly produce fractures within which the "high sulfate" form of calcium sulfoaluminate precipitates as prismatic or acicular crystals. In fact, even prior to development of fractures, accumulations of fibrous and subspherulitic groups of crystals may develop in air voids disseminated throughout the cement paste. It is to be noted that the formation of the compound plays no essential rôle in the expansion of concrete affected by alkali-aggregate reaction (**16**).

In answer to Mr. Sprague's question, it is to be noted that, according to available data, those deleterious materials which cause a reduction in alkalinity greater than about 225 millimoles per liter in the described chemical test so reduce the availability of cement alkalies that mortar expansion is negligible when these materials constitute the entirety of the aggregate. In other words, the "pessimum" proportion for these materials is low (2 to 20 per cent); and although the mortar expansion typically is high at the "pessimum" proportion, expansion is decreased greatly by further addition of the materials to the aggregate. Consequently, Fig. 1 of the paper might with benefit be modified by separating the data points representing deleterious materials by a line crossing the right-hand field of the graph at about 225 millimoles of alkalinity reduction (Fig. 30). The materials which are innocuous when constituting the entirety of the aggregate include: No. 59, siliceous magnesian limestone; No. 31, a rhyolite tuff; No. 57, opal; No. 32, a rhyolite; No. 49, opaline chert; No. 85, a deeply altered rhyolite tuff composed essentially of heulandite and montmorillonite; No. 74, an opaline shale; and No. 75, opaline shale No. 74, after calcination.

---

[6] Director, Division Materials Testing Lab., Corps of Engineers, U. S. Department of the Army, Marietta, Ga.

[7] W. Lerch, F. W. Ashton, and R. H. Bogue, "The Sulphoaluminates of Calcium," *Journal of Research*, Nat. Bureau Standards, Vol. 2, pp. 715-731 (1929).

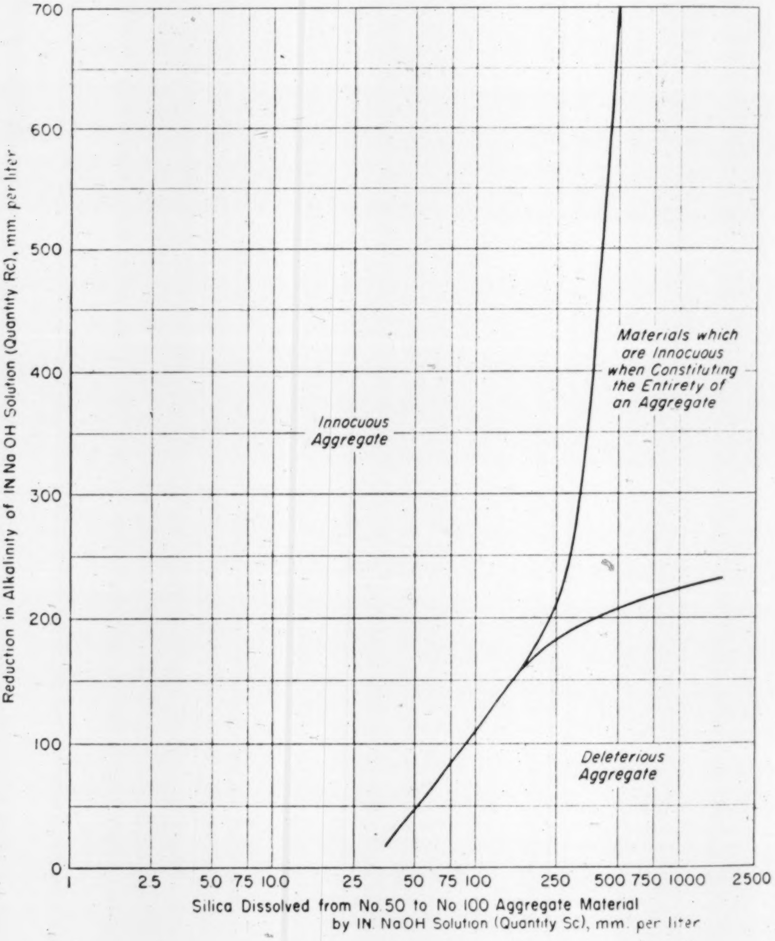


FIG. 30.

Unfortunately for simplicity, not all of the deleterious materials effecting an alkalinity reduction less than 225 millimoles per liter cause a deleterious degree of mortar expansion when they constitute the entirety of the aggregate. Aggregates No. 51, a flint; No. 53, a chalcedonic chert; and No. 54, chalcedony, all of which are characterized by a "pessimum" proportion of 20 per cent and at the "pessimum" proportion cause mortar expansion of 0.137 per cent to 0.264 per cent linearly at 1 yr., cause mortar expansion of merely 0.006 per cent to 0.028 per cent linearly at 1 yr. under the same conditions when they constitute the entirety of the aggregate in the mortar. The available data suggest that deleterious materials causing maximum mortar expansion when they constitute less than 20 per cent by weight of the aggregate typically cause only insignificant mortar expansion when constituting the entirety of the aggregate. Deleterious materials whose "pessimum" proportion is more than 20 per cent typically cause significant mortar expansion when they constitute the entirety of the aggregate. If the "pessimum" proportion is 20 per cent, then the mortar expansion may be large or negligible when the material constitutes the entirety of the aggregate.

In view of these considerations, it would appear possible that the aggregates described by Mr. Sprague, which cause high alkalinity reduction and high silica release in the test, might not have caused excessive expansion of structures because, as used, they greatly exceeded the "pessimum" proportion. On the other hand, one must always bear in mind that they might be innocuous in those situations because they were used with low-alkali cement, or for other reasons, as discussed in the original paper.

A RAPID METHOD OF TESTING MATERIALS FOR THE ALKALI-AGGREGATE REACTION*

By D. O. Woolf[1] and Theodore R. Smith[1]

The generally accepted method of testing aggregate for reaction with the alkali in portland cement follows suggestions made by T. E. Stanton in 1941.[2] In this method, the aggregate is used to prepare mortar bars, and the change in volume of these bars is determined at periodic intervals. With extremely reactive materials, a decision regarding the use of the material with a given type of cement may be made in a few months, but usually the tests must be continued for one or more years.

This method of test has several disadvantages, possibly the most serious being the time usually required to test a material other than one which is extremely reactive with the alkali. When this time exceeds a month, as it usually does, the method becomes unsuitable for the routine or acceptance testing of aggregates. When samples of coarse aggregate are prepared for test by crushing the material to sand size, all of the crushed material customarily is used in preparing the mortar. In early studies of alkali-reactive aggregates, it was found that the size of the reactive aggregate had a marked effect on the amount of expansion. If the reactive aggregate was crushed fine enough, the mortar would develop little if any expansion. This same behavior may be found in tests of crushed aggregate when finely crushed fragments are included in the sample tested. Other disadvantages of the method include the fragility of the bars (especially when the smaller bar is used) and the difficulty of maintaining a saturated atmosphere for storing the bars. A method frequently used for storing the bars consists of placing the bars above a pool of water in a metal container, and sealing the cover. With a long storage period between consecutive inspections of the bars, and the use of galvanized containers, the loss of water and subsequent drying of the specimens by the development of pin holes in the container has frequently been reported.

In an attempt to accelerate the reaction between the alkali in the cement and the aggregate, the addition to the mortar of 1 per cent of sodium hydroxide by weight of the cement has been tried. In some instances, mortar so treated has been found to have as much expansion at an age of 3 months as plain mortar developed in a year. Unfortunately, for some reason which has not been determined, a satisfactory correlation between the two methods has not been established. Too frequently, a treated mortar prepared with a high-alkali cement will show an excessive amount of expansion at an early age, but plain mortar prepared with the same cement will develop only a small amount of expansion at considerably greater ages. In contrast to this, repeated tests have shown that when a low-alkali cement is used with a reactive aggregate, the addition to the mortar of 1 per cent of sodium hydroxide, by weight of the cement, does not change materially the amount of expansion developed by the mortar. In spite of the lack of agreement between the results obtained with the long-time and accelerated tests, the use of mortar treated with sodium hydroxide has been found of value in furnishing at an age of 3 months information as to whether the material under test is unlikely to develop excessive expansion at some greater age when tested in plain mortar. If the treated mortar prepared with high-alkali cement fails to show excessive expansion at an age of 3 months, the aggregate used may be considered to be relatively nonreactive. However, if the mortar shows high expansion in the accelerated test, no definite conclusion can be drawn.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Senior Materials Engineer and Senior Scientific Aid, respectively, Division of Physical Research, Public Roads Administration, Washington, D. C.

[2] T. E. Stanton, "The Expansion of Concrete Through Reaction Between Cement and Aggregate," *Proceedings*, Am. Soc. Civil Engrs., Vol. 67, p. 1402 (1941).

In further search for an accelerated method of test for the alkali-aggregate reaction, a method proposed by Alderman, Gaskin and Vivian was studied.[3] This method involves the warping of a slab of mortar due to differential expansion between the two mortars, one containing nonreactive aggregate and the other containing the aggregate under test, with which the specimen is prepared. In the original report the authors state that the "bi-aggregate slabs have not failed to indicate expansive reaction in mortars which at atmospheric temperatures have developed significant expansions in twelve months," and that "in no instances have bi-aggregate slabs given indications of expansive reaction which were not fully substantiated by other observations." The mention of "atmospheric temperatures" is understood to refer to the room temperatures (58 to 59 F.) used in the tests at Melbourne. Tests made by the Public Roads Administration do not support these statements fully. Many instances have been found where reactive mortars, as determined by other tests, have failed to develop the spotting or warping which are stated to be indicative in the bi-aggregate slab test. In other cases, very severe spotting has developed on slabs prepared with a mortar which, to an age of 3 yr., has shown only an immaterial amount of expansion in bar tests. These slabs did not warp, and the question arises whether the spotting itself may be considered to indicate the alkali-aggregate reaction. When the indication of the reaction was limited to marked warping of the slab, the test was found to be no more rapid than the accelerated bar test in which the mortar was treated with sodium hydroxide. Of these two tests, the latter was preferred due to its quantitative determinations.

The use of large numbers of aggregates obtained from noncommercial sources in the western part of the country caused some thought to be given to the development of a method of test for alkali-aggregate reaction which could be made with a minimum of testing equipment and with no requirements for delicate or expensive apparatus. The development of such a test would permit the examination of samples of aggregate in field offices and reduce the number of samples submitted to the laboratory for more thorough tests. In an effort to develop this test, some study was given to the preparation of a hard and brittle container of mortar to enclose the mortar prepared with the aggregate under test. This was on the assumption that, should the mortar under test develop expansion due to chemical reaction, the expansion would crack the external mortar shell and reveal the reactive character of the aggregate at an early age. Difficulties were found in the preparation of the hard mortar containers. Although a hard mortar could be obtained with a 1:2 mix and a water-cement ratio of 0.25, the mortar had to be tamped in place and specimens of uniform density were seldom obtained.

---

[3] A. R. Alderman, A. J. Gaskin, and H. E. Vivian, "A Qualitative Test for Cement-Aggregate Reaction," *Journal for the Council for Scientific and Industrial Research* (Melbourne, Australia), Vol. 18, No. 4, November, 1945, p. 433.

To furnish a hard and brittle container for samples of mortar under test for alkali activity, the use of Mason jars was suggested and tried initially by the junior author. These proved quite successful and a large program of tests to determine the suitability of the method was begun. The mortars included in the first series of tests were prepared with known reactive and nonreactive aggregates, and with both high- and low-alkali cements. The jars used had various shapes of horizontal cross-section. In some, the cross-section was circular; in others the cross-section was square with rounded corners of short radius; and in a third type the radii of the rounded corners were so large that the cross-section approached the circular. Some jars were filled to the neck; others were filled about half full. After the mortar had been placed in the jar, the jar cap was fastened and the jar set aside until the mortar hardened. A small amount of water, about 25 ml., was then added to each jar, and the cover replaced. Later in the study of the method, it was suggested that the jars be inverted after the water had been added to insure that the alkali in the mortar would not be leached out. This practice was followed, not necessarily to prevent leaching of the alkali, but to permit more ready detection of cracking in the jar.

Some disappointing results were obtained in the first series of tests. A large number of jars containing mortar prepared with high-alkali cement and a nonreactive aggregate failed at an early age. Other jars containing reactive mortar did not fail within a reasonable length of time. Some jars cracked, while others containing the same mortar did not. The failure of the jars containing a high-alkali cement and an inert sand was traced to a reaction between the cement and the glass of the jar. This was subsequently prevented by coating the jar with an alkali-proof paint. A number of coating materials were tried, but best results were obtained with pigmented materials having a chlorinated rubber or a vinyl resin base. Of these two, that with the chlorinated rubber base is preferred. The coating material with the vinyl resin base seems to be tougher and less liable to damage when the jar is being filled, but that with the rubber base has a less objectionable odor, is easy to handle, and dries very rapidly. Several different colors of the rubber base finish were tried, including white, black, and gray. The white coating is not desirable as it interferes with the detection of the dried gel, the end product of the alkali-aggregate reaction, when the mortar is removed for inspection from the jar after failure.

The mechanical strength of the jars undoubtedly has a considerable influence on the results of this test, and the variable results obtained in the first series of specimens possibly were due to the use of different types of jars of variable strength. From observation of the tests results obtained, the least desirable type of jar appeared to be that with a square cross-section and rounded corners of short radius. Many jars of this type failed only at the junction between the sides and the bottom, and it appears that this is the weakest point in the jar. Jars with a truly round cross-section appeared to give the most uniform results, but difficulty was found in obtaining these. The jars most readily available seem to be the square jar with short radius corners and the jar with heavily rounded corners and a cross-section approaching the circular. Some jars used in these tests contained striations or mold marks in the glass, and an attempt was made to obtain a jar of more uniform quality. Consideration was given to glass jars used for packaging food stuffs, such as mayonnaise, and these jars were compared for uniformity with the Mason jars of nearly circular cross-section. The results obtained with the two types of jar differed so little that it was decided to adopt the latter for use in the test, or the round Mason jar when it could be obtained. These investigations indicated that it would be desirable to make more than one jar specimen for each mortar, and the practice of preparing three jars of a kind was adopted. The use of smaller containers such as shell vials was tried and found satisfactory in tests of mortars, but the pint Mason jar is preferred as this can be used for testing concrete with a maximum size of aggregate of at least 1 in.

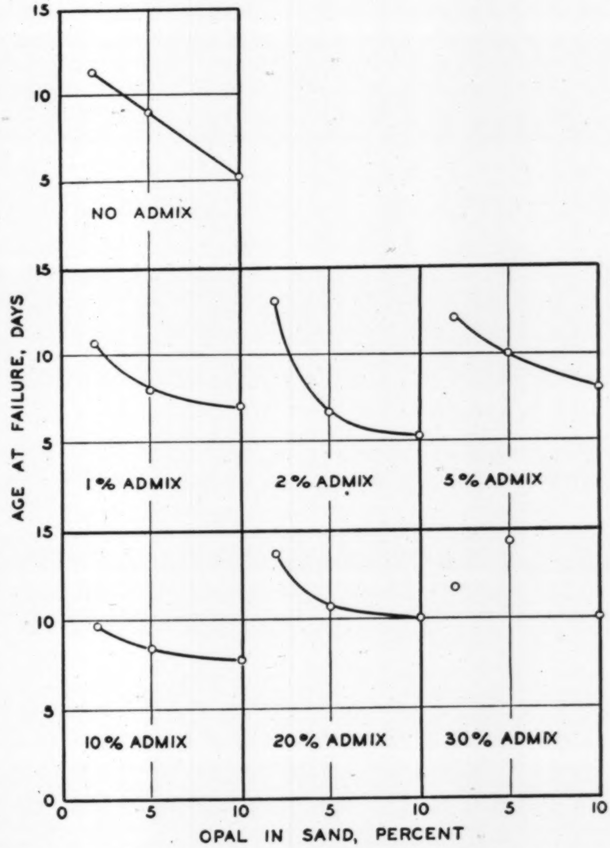


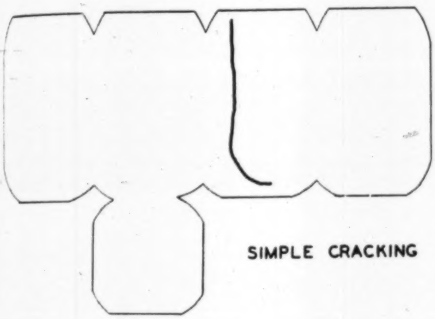
FIG. 1.—Example of Use of Mason Jar Test Showing Per Cent of Opal in Sand.

Storage of the jars at room temperature has been the general practice in most tests conducted to date as this condition probably would be followed in tests in the field. Some tests were made at 120 F. to determine the effect of the higher temperature. Included in this group were test specimens for a number of gravels composed essentially of quartz and chert. Many of these jars broke at a very early age, in several cases at only 2 or 3 days, and it was suspected that the cracking of the jar might have been caused either by increase in volume of the aggregate due to absorption of water, or to expansion of the mortar or concrete due to heat. To investigate these possibilities a series of tests was made on samples of concrete prepared with high-alkali cement, quartz sand, and a gravel containing chert or a crushed granite. Both coarse aggregates were graded between the 1-in. and No. 4 sieves. A batch of concrete, sufficient for six jars, of 1:2:3 mix was prepared, with each coarse aggregate in an oven-dry or saturated condition. Three of each group of six jars were stored at laboratory temperature until the concrete hardened. The other three were placed in a cabinet held at 120 F., as soon as they were prepared. At an age of 24 hr., water was added to all jars, and those which had been stored at laboratory temperature were placed in the warm storage cabinet. The jars were inspected daily for the development of cracking and the test continued to an age of 71 days.

The results obtained in this series of tests indicate that failure of the jars may be caused entirely by volume changes due to absorption of water by certain coarse aggregates, or by the thermal volume change of hardened concrete. Consequently, coarse aggregates used in this test should be saturated prior to mixing if they contain types of aggregate which are highly absorptive. Included in these types of aggregates are chert and some varieties of limestone. If the jars are stored at an elevated temperature, they should be placed in this storage before the content of the jar has hardened. It should be stated that in tests of fine aggregate, that is, material passing the No. 4 sieve, no difficulty due to failure by thermal volume change has been found.

The Mason jar test is being used in studies of the effect of an admixture of a finely divided siliceous material on the alkali-aggregate reaction. In a preliminary investigation concerned primarily with the amount of this admixture to be used in a reactive mortar, the results given in Fig. 1 were obtained. This figure is presented to show that quantitative results can be obtained with this method of test. All specimens were prepared with a high-alkali cement and all contained opal except one group of reference specimens. Each point in Fig. 1 represents an average of three specimens. The tests were discontinued at an age of 30 days, when all except the reference specimens had failed. With few exceptions, the three jars in each group gave consistent results. In the entire set of 21 groups containing reactive aggregate, the three jars in each of 14 groups broke at the same age or at an age of within one day of the average for the three jars. In only four groups did any of the jars break at an age more than two days from the average. As all jars developed the same type of cracking, it is believed that a marked difference between the ages of failure of specimens of a group may be due to differences in strength between the jars. Methods for classifying jars prior to use are being considered.

The test is considered of most value for the rapid determination of the susceptibility of aggregates to alkali and for the determination of the compatibility of given combinations of cement and aggregates. The test specimens are prepared readily with very simple equipment. Although a balance has been used to weigh the components of the mortar or concrete, proportioning of the mix by volume should be satisfactory. For 1:2 mortars, a water-cement ratio of 0.5 by weight, or 0.75 by volume, has been found suitable. When it is desired to test concrete mixtures, sufficient water to give a plastic mix should be used. The prepared mortar or concrete is placed in the coated jar with care to prevent scratching the coating, and the bottom of the jar is struck several times with the palm of the hand to settle the contents. The jar is then capped. At an age of 20 to 24 hr., about 25 ml. of water is added to the jar, the cap replaced firmly, and the jar placed in storage. Daily inspection of the jars is advisable. When the jars are stored in an inverted position, the cracking frequently can be seen without moving the jar if the bottom and an inch or so of the side of the jar can be inspected.



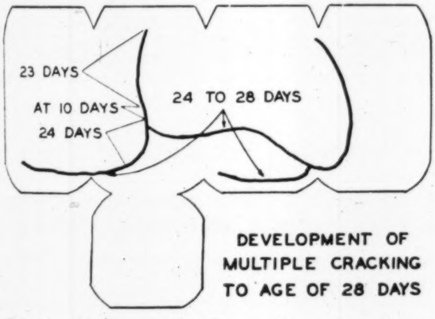


FIG. 2.—Failures of Mason Jars—Developed Views of Sides and Bottom of Jars.

Various forms of cracking are found. Sometimes a simple vertical crack will appear. If this cracking does not develop further, it is considered to indicate an expansion of very low order resulting possibly from an alkali-aggregate reaction which may be dismissed as immaterial. However, if simple cracking progresses into multiple cracking, or as is most often the case, the jar develops multiple cracking at the initial failure, the failure may be considered to indicate an aggregate of high susceptibility to reaction with the alkali in cement. Sketches of jars showing simple cracking, simple cracking which developed into multiple cracking, and initial multiple cracking are shown in Figs. 2 and 3.

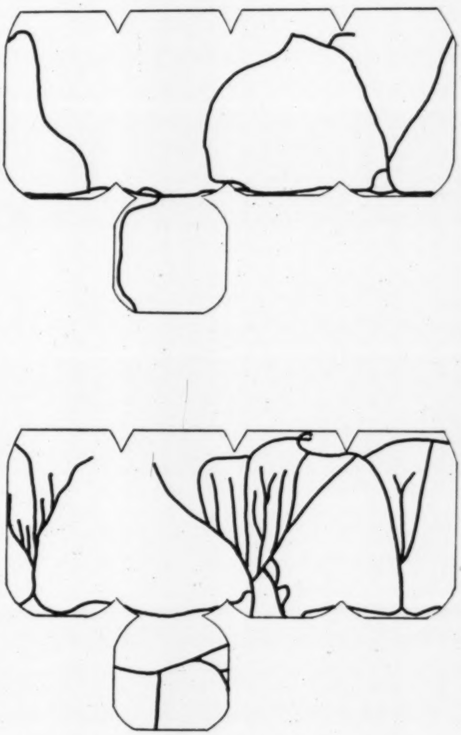


FIG. 3.—Multiple Cracking of Mason Jars—Developed Views of Sides and Bottom of Jars.

CONCLUSION

A simple and rapid method of testing mortar or concrete for the alkali-aggregate reaction is furnished by the Mason jar test. A period of test of 28 days appears to be sufficient for the detection of aggregates which may be considered too susceptible to reaction with high-alkali cement to warrant their use in concrete. The method is suitable for use in the field as the equipment necessary is inexpensive and can be obtained readily.

DISCUSSION

Mr. H. S. Meissner.[1]—I visualize that as you screw the cover on the jar you must be exerting some preliminary pressure. You probably use the rubber seal. Do you have some means of letting out air, making sure that the entire volume is completely filled with solids or liquids?

Mr. D. O. Woolf (*author*).—The jar is usually filled within an inch of its top. No attempt is made to bleed the jar of pressure developed by its closure.

Mr. Meissner.—There is air there too, then? You are getting the expansion only in the sides and towards the bottom; it is free to expand downwards, since the jar is inverted.

Mr. Woolf.—There is usually some air in the jar. The mortar or concrete is free to expand downward.

Mr. Meissner.—But the curve of the jar is sufficient to confine it, then, so that the pressure all goes toward cracking?

Mr. Woolf.—Tests made with jars both half-full and filled to or above the neck have failed to show any significant differences in the time or amount of cracking.

Mr. Stanton Walker.[2]—The National Sand and Gravel Association Research Foundation, in cooperation with the Research Laboratory of the National Ready Mixed Concrete Association, both at the University of Maryland, has done a limited amount of work with a "Mason-jar test" similar to that described by Messrs. Woolf and Smith for detecting reactions between alkalies of cement and certain constituents of the aggregate. We share the authors' dissatisfaction with the uncertainty and time consumption of conventional tests. The need for more rapid and more nearly qualitative tests is clearly indicated. However, our experiences do not appear to have been as favorable as those of the authors, which may very well be due to our neglect of certain refinements which they took into account.

Our work with Mason jars was done as a part of our studies of the reactivity of certain combinations of sand and gravel in concrete. The approach was suggested to us by Mr. W. F. Abercrombie, Engineer of Tests of the Georgia Highway Department at a time when he accompanied us on an inspection of Georgia highway structures. The tests were carried out and the data analyzed and summarized by Fred F. Bartel, Assistant Director of Engineering, D. L. Bloem, Associate Research Engineer, and E. J. Ziegler, Assistant Research Engineer.

To supplement information reported by the authors, we are summarizing herein the results of our tests. A total of 72 Mason jar specimens was molded. Three combinations of aggregates were used as follows:

1. Sand and gravel, predominately quartz, from near-by Maryland, used as "stock" aggregates in our laboratory.
2. Crushed trap rock from Connecticut and a natural calcareous sand from Ohio.

---

[1] Research Engineer, U. S. Bureau of Reclamation, Denver, Colo.

[2] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

3. Sand and gravel from a southeastern state, predominately quartz, but containing, as an impurity, considerable chert in various stages of disintegration.

Both the sand and gravel of the first combination of materials—not from the same deposit—are considered to have an excellent service record. No indications of alkali reactivity attributed to them have been called to our attention, and they are used in an area where cements relatively high in alkali are likely to be encountered. The trap rock and calcareous sand were chosen as most unlikely to be reactive. The third combination was selected because of its record in the field—good with certain cements and not so good with others.

The aggregates were well-graded up to 1 in., with the sand and gravel combinations having a fineness modulus of 4.9 and the sand trap combination 4.8. The water-cement ratio was 6.9 gal. per sack and the slump was estimated (the batches were too small to measure it) to be 3 to 4 in. The cement factor was computed on the basis of absolute volume to be 5.5 sacks per cu. yd. of concrete.

Two cements were used—one classified as low in alkali and the other as high. While we did not determine the alkali content for these samples, routine tests of others indicate the cement from the low-alkali source to be well under 0.5 per cent (about 0.45 per cent) and from the high-alkali source approximately 0.8 per cent or somewhat more.

Three batches of concrete were made at different times (that is, three rounds) for each of these six conditions (three aggregates combinations and two cements) and four Mason jar specimens were made from each batch and treated as follows:

1. Jars sealed with rubber and lid and, after standing overnight at room temperature, stored at 100 ± 5 F.

2. Jars coated with transparent rubber base paint (Paracoat), sealed, and after standing as before, stored at 100 ± 5 F.

3. Jars coated as in 2, not sealed (lid turned on loosely) and, after standing, stored at 100 ± 5 F.

4. Jars coated, sealed and stored at 75 ± 5 F.

The jars were observed daily for the first week and at increasing intervals thereafter until eight weeks when observations were discontinued. The conditions of the jars were rated in three ways as follows:

1. Relative cracking at two weeks.
2. Relative cracking at eight weeks.
3. Relative age at which cracking started.

In the first two methods of rating, 100 per cent represents the maximum degree of cracking in each case while, for the third, ratings are, roughly, the complement of the percentage which the age at first crack is of the maximum age—that is, eight weeks. Thus, to make the relationship in the same direction as the first two, a jar going the entire period of eight weeks without cracking was rated as 0 per cent while one cracking early (arbitrarily, less than four days) was rated as 100 per cent. In the first two cases, numerical values were obtained by assigning numbers to degrees of cracking judged as "very bad," "bad," "moderate," etc.

The average of the ratings for the three batches for each of these three bases are shown in the accompanying Table I. While values for individual specimens are not given, a study of the data shows them to be reasonably uniform and of about the same order of magnitude. Note also, in Table I, that averages combining the three bases of ratings are given.

In addition to the ratings just described, the jars were examined for any visible evidence of chemical action.

There can be no doubt but that some took place. A transparent ooze was exuded through the cracks of several of the jars. When the glass jars were broken away from the concrete, the surfaces of the specimens were speckled with white, chalky craters similar to those generally associated with alkali aggregate reaction. Further, chemical analyses of the ooze, made through the courtesy of the Public Roads Administration, indicated it to consist of about 25 per cent water, 25 per cent silica, and 50 per cent sodium or potassium oxide, together with small amounts of calcium, magnesium, and miscellaneous oxides.

TABLE I.—SUMMARY OF DATA OF MASON-JAR TEST FOR ALKALI REACTIVITY.

Aggregate graded 0 to 1 in.; concrete contained 6.9 gal. water per sack of cement and approximately 5.5 sacks of cement per cu. yd.

Concrete mixed and stored at approximately 80 F. overnight before being stored at 75 or 100 F.

Each value is the average of 3 separate tests.

Method for Rating Expansion	Expansion as Indicated by Cracking of Jars							
	Low-Alkali Cement				High-Alkali Cement			
	Local Quartz Gravel and Sand	Crushed Trap Rock and Calcareous Sand	Southeastern Quartz Gravel and Sand	Avg.	Local Quartz Gravel and Sand	Crushed Trap Rock and Calcareous Sand	Southeastern Quartz Gravel and Sand	Avg.
Aggregate Combination	(1)	(2)	(3)		(1)	(2)	(3)	
STORAGE IN UNCOATED MASON JARS TIGHTLY SEALED, AT TEMPERATURE 95 TO 105 F.								
Relative cracking at 2 weeks	50	11	56	39	100	56	94	83
Relative cracking at 8 weeks	81	62	81	75	100[c]	76	95[b]	90
Relative age when jar cracked	67	52	67	62	100	71	100	90
Avg.	66	42	68	59	100	68	96	88
STORAGE IN COATED MASON JARS TIGHTLY SEALED, AT TEMPERATRE 95 TO 105 F.								
Relative cracking at 2 weeks	50	0	11	20	56	22	50	43
Relative cracking at 8 weeks	71[a]	29	48	50	52[b]	38	52	47
Relative age when jar cracked	90	19	48	52	81	57	76	71
Avg.	70	16	36	41	63	39	59	54
STORAGE IN COATED MASON JARS LOOSELY SEALED, AT TEMPERATURE 95 TO 105 F.								
Relative cracking at 2 weeks	0	0	11	4	33	6	50	30
Relative cracking at 8 weeks	29	33	38[b]	33	52	29	62[a]	48
Relative age when jar cracked	28	19	38	28	81	43	81	68
Avg.	19	17	29	22	55	26	64	49
STORAGE IN COATED MASON JARS TIGHTLY SEALED, AT TEMPERATURE 70 TO 80 F.								
Relative cracking at 2 weeks	0	0	0	0	0	0	0	0
Relative cracking at 8 weeks	19	0	5	8	19	19	29	22
Relative age when jar cracked	14	0	5	6	33	28	28	30
Avg.	11	0	3	5	17	14	19	17
GRAND AVERAGE ALL STORAGE CONDITIONS								
Relative cracking at 2 weeks	25	3	20	16	47	21	48	39
Relative cracking at 8 weeks	50	31	43	42	56	40	60	52
Relative age when jar cracked	50	22	40	37	74	50	71	65
Avg.	42	19	34	32	59	37	60	52

[a] Exudations found on two specimens.
[b] Exudations found on one specimen.
[c] Exudations found on three specimens.

Of the twelve specimens showing ooze, all but three were made with the high-alkali cement and all were made with the quartz sands and gravels. Only five of the twelve came from the suspect southeastern sand and gravel, the remaining seven being from the local quartz sand and gravel. Seven of the twelve specimens showing ooze were in coated jars and all were in jars stored at 100 F.—the 75 F. storage resulting in no exudations. (It is pertinent to interject that beam specimens, made with these same materials, have shown the ooze and white, chalky deposits but no significant changes in length as measured by a strain gage over a 10-in. gage length.)

So far as the jar ratings are concerned some significant trends are indicated, although clear-cut relationships are not shown. These trends may be summarized as follows:

1. With only one exception, the concrete made with the high-alkali cement showed significantly higher expansion (as indicated by more or quicker cracking) than that made with low-alkali cement. For the grand average of all results the severity may be expressed approximately at the ratio of 52 to 32. The evidence seems plain that the cement did enter into the picture, although it is not entirely clear that it was the alkali content which represented the significant difference. It should not be overlooked however that the distribution of the exudations lend force to the belief that alkali content entered into the picture.

2. The siliceous aggregate, combinations 1 and 3, showed significantly higher volume changes than the trap-calcareous sand combination. The higher thermal coefficient of expansion of the quartz might offer a satisfactory explanation for these differences, although, here again, one is led to suspect that alkali reaction entered into the picture.

3. The coated jars showed less cracking than the uncoated ones. This may have been because the coating prevented any reaction between the cement and the glass, or, it tended to cushion the effect of the volume changes of concrete due to temperature, moisture, hydration, etc., or, it broke the bond between glass and concrete. However, it is evident that specimens with the high-alkali cement were more improved by the coating than those with the low-alkali cement, which suggests the possibility that chemical reaction with the glass might have been involved.

4. With certain exceptions, the unsealed jars showed less volume change than the sealed ones. This might be explained by greater drying and hence less expansion, or, by some relief in pressure due to loss of air.

5. The two siliceous aggregates gave substantially the same results.

While these trends are interesting, and probably significant, they would not appear to support the conclusion of Messrs. Woolf and Smith that:

> "A simple and rapid method of testing mortar or concrete for alkali reaction is furnished by the Mason-jar test. The period of test of 28 days appears to be sufficient for the detection of aggregates which may be considered too susceptible to reaction with high alkali cement to warrant their use in concrete. The method is suitable for use in the field and the equipment necessary is inexpensive and can be obtained readily."

On the other hand, as stated at the outset, our failure to secure data justifying so firm a conclusion may have been due to our neglect of certain refinements. Among the differences in our procedure and that used by the authors, which may be significant are:

1. Our specimens were stored overnight at room temperature and then removed, in an upright position, to a cabinet maintained at a temperature of 100 ± 5 F. or, as indicated, to a room maintained at about 75 ± 5 F. The authors, on the other hand, kept their

specimens at room temperature throughout the test; on the day following molding, water was added to the small volume remaining at the top of the jar, the jar was capped and inverted.

2. In our tests ordinary 1-pint cylindrical Mason jars were used with no attempt at selection. The authors, on the other hand, attempted to select jars free of strain.

3. Our jars were coated with a transparent paint while the authors used a pigmented rubber base paint; the latter may have permitted of more ready detection of imperfections in coatings than the former.

Whatever the significance of these differences, I think it doubtful if we have arrived at the point where we can assign to a field test so important a responsibility as determining if aggregates are "reactive."

MR. WOOLF (*author's closure by letter*). —It is regrettable that Mr. Walker chooses to condemn a method of test on the basis of a single group of test specimens made, as he admits, under possibly questionable procedures. The test results which he obtained are similar to those found in early tests by the Public Roads Administration. If the general trend of the test results obtained by Mr. Walker is overlooked for the moment, it appears quite possible that these results were influenced in detail by the use of old jars, by failure to coat the jars thoroughly, and by storage of the specimens at a higher temperature after the concrete had hardened.

One other feature which has not been given proper consideration should be mentioned. The "low-alkali" cement used by Mr. Walker is stated to have an alkali content of about 0.45 per cent. In a discussion of a paper on distress of concrete in Georgia,[3] the writer presented some information of the behavior of portland cement which had been obtained by the Public Roads Administration. In these tests, the expansion due to reaction with aggregate of 51 samples of portland cement varying in alkali content from less than 0.2 per cent to more than 1.0 per cent was determined. Ten samples of cement had alkali contents between 0.41 per cent and 0.50 per cent. Mortar prepared with four of these ten samples developed enough expansion at the age of one year to classify the cements as highly reactive with susceptible aggregate. Apparently the controlling factor is not the total amount of alkali in the cement but is the availability of the alkali for chemical reaction.

In the tests reported by Mr. Walker, the cement described as "low-alkali" may have been of the type in which the alkali is readily available for chemical reaction. If so, the results reported can be understood more readily than they could be with the incorrect assumption that an alkali content of 0.45 per cent invariably indicates a cement free from objectionable characteristics with respect to chemical reaction with aggregates. With this understanding, the general trend of the test results reported by Mr. Walker shows the Mason jar test to be suitable for the detection at early ages of undesirable combinations of cement and aggregates.

---

[3] W. F. Abercrombie, "Concrete Deterioration in Georgia Associated with Alkali-Reactive Aggregates," *Proceedings*, Southeastern Assn. State Highway Officials, Sixth Annual Meeting, p. 80 (1947).

PETROGRAPHIC IDENTIFICATION OF REACTIVE CONSTITUENTS IN CONCRETE AGGREGATE[*][1]

By Bryant Mather[2]

The microscope has been employed for the study of rocks in thin sections with polarized light for more than 100 yr., and the immersion method has been used in the examination of mineral particles for about 50 yr. During this time these techniques have become highly refined and a large amount of data has been developed. The intensive application of the techniques and data of petrography to research in concrete and concrete aggregates began only about 10 yr. ago at about the time that interest developed in the problems of deleterious chemical reactions in concrete. In part, these two developments coincided fortuitously but, in part, they were causally connected. At the present time, the literature of petrography is well supplied with information on the use of techniques and determinative criteria for the identification of virtually all mineral species, rocks, and other materials which may be encountered in concrete aggregates. The applications of these techniques and criteria to the identification of reactive constituents in concrete aggregate therefore required no development of new methods but merely the development of data as to which of the constituents of concrete aggregates were reactive. Coombs in 1942 **(1)**[3] stated the problem in these words: "The ultimate goal is to devise some easy way to detect the reactive aggregate before it is used in concrete. To do this, however, it is first necessary to find out what rocks or minerals are reacting with the cement." This has been done in a considerable number of cases. For example, the disintegration of concrete made with what has been referred to as "siliceous magnesian limestone" and related materials was described by Stanton in 1944 **(2)** as follows: "It was definitely determined that the cause of the trouble in California was the opaline silica in the chert." Other examples will not be cited here since the subject has recently been thoroughly summarized by McConnell, Mielenz, Holland, and Greene **(3)**. These authors state (p. 127): "Opal, chalcedony, tridymite, intermediate to acidic volcanic glasses, and probably the hydromicas of some phyllites, are recognized as deleterious constituents of concrete aggregates."

When any one of these reactive constituents is present in a material used as aggregate in concrete, the result to be expected is a deleterious chemical reaction. The possibility that the expected reaction will not occur or that it may be forestalled—as a result of proper attention to the chemical composition of the cement, the use of an inhibitor, the fact that the deleterious constituent is present in an amount much greater than or much less than the "pessimum" amount, or the favorable location and exposure of the structure, should always be borne in

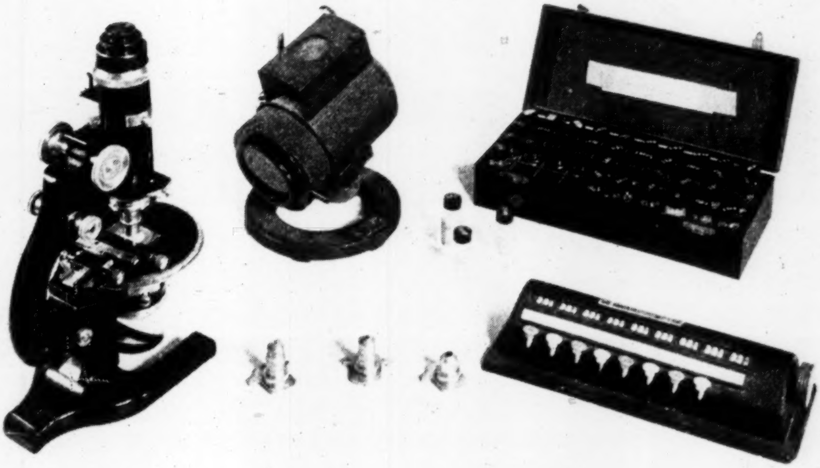
---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

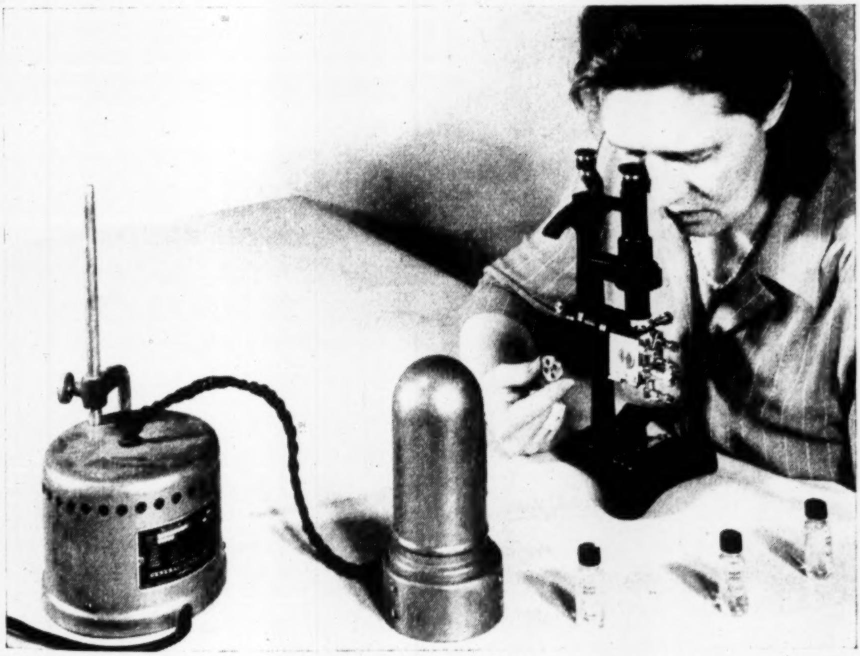
[1] Approved for presentation and publication by the Office, Chief of Engineers.

[2] Engineer (Concrete Research), Concrete Research Division, Waterways Experiment Station, Mississippi River Commission, Corps of Engineers, Clinton, Miss.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1124.



(*a*) Petrographic Microscope, Set of Immersion Media, and Counter.



(*b*) Sodium Arc Lamp and Abbé Refractometer Used in Calibration of Immersion Media.

FIG. 1.—Apparatus Used in Petrographic Identification of Reactive Constituents in Concrete Aggregate

mind by the petrographer when called upon to predict what *will* happen if a certain aggregate is used, and by the engineer who evaluates such predictions. However, in the present state of our knowledge these are mere possibilities—none can be counted on to give a safe guarantee of freedom from trouble if reactive materials are in the aggregate.

It is therefore essential to have means of positively detecting such constituents. Mielenz in 1946 (4) in a paper entitled "Petrographic Examination of Concrete Aggregates," described the four materials listed by McConnell, Mielenz, Holland, and Greene as "deleterious" as "known to react deleteriously with high-alkali cements" (p. 311), but gave no techniques or criteria for distinguishing them from similar or related substances. These materials can, however, be positively identified and distinguished from related substances by the standard techniques of petrography, particularly by examination of particles using the immersion method to determine relative index of refraction. The determination of index of refraction by the immersion method involves the equipment shown in Fig. 1 (*a*), consisting of a petrographic microscope, a light source, and vials of immersion media. The counter, which was designed for use in counting blood cells, is optional but has been found to be very convenient. Since some of these determinations must be quite accurate it is necessary frequently to calibrate the immersion media. Figure 1 (*b*) shows this operation being performed using an Abbé refractometer and a sodium-arc lamp as a source of monochromatic illumination.

The aggregate constituents that are known to be deleteriously reactive are described and diagnostic procedures and criteria for identifying them are outlined:

Opal:

Opal is a material which may occur in igneous, sedimentary, or metamorphic rocks but is always formed by deposition from aqueous solution and therefore when found in volcanic rocks it is of secondary origin. It often occurs in the form of veinlets, cavity fillings or linings, or as a replacement of organic materials, as in petrified wood, and is found at many localities as the cementing material in sandstone. Chemically, it consists of amorphous silica with a variable amount of water. As the water content varies, its index of refraction and density varies. However, its index of refraction is usually about 1.45 and seldom higher than 1.46 or lower than 1.40. The low index and amorphous character of opal serve to distinguish it. In some cases, its presence may at first be overlooked, particularly during the examination of thin sections, since it may be difficult to distinguish a particle or veinlet of opal from a hole or crack in the thin section. Careful examination, however, will prevent its being overlooked for these reasons.

Chalcedony:

It has been indicated by a considerable number of investigators that chalcedony is *not*, as it is described in many reference books which are still in use, merely a descriptive term for the microfibrous form of quartz. That this obsolete concept dies hard is illustrated by the fact that as recently as December, 1947 the author of an abstract (5) in the *Bulletin* of the Geological Society of America stated: "Arguments are advanced in favor of the idea that chalcedony is a distinctive mineral and not merely a variety of quartz." It is true that the principal—and only crystalline—constituent of chalcedony is low-temperature quartz in microfibrous form; however, the index of refraction and density of chalcedony when considered from the standpoint of form birefringence reveal that it contains an interstitial material between the fibers and that this interstitial material must be a substance having all of the properties of opal. These relationships were cited by the author in 1945 in a discussion (6) designed to elucidate a published report in which the reactivity of chert and chalcedony appeared to surprise the authors. In this connection it is noted that the petrographic work referred to in the paper discussed at that time did not involve the criteria referred to below, and the separation of quartz and chalcedony was based on textural rather than physical or optical criteria (7). In this connection I should like to call attention to one sentence from a proposed definition of chert considered by Subcommittee II-f of Committee C-9: "It (chert) is composed of quartz, chalcedony, or opal, or mixtures of these minerals, with chalcedony as the dominant mineral in most cherts."

The term "chert" is, it is believed, properly used as a term to describe particles which upon megascopic inspection are found to consist of fine-grained white or colored material which appears to be silica and which characteristically has a conchoidal fracture. The term, used in this way would include fine-grained quartz; chalcedony; many varieties of fine-grained silica the names of which are based on color or texture such as jasper, agate, hornstone, etc.; and, in some cases, opal. The separation of chert into groups based on whether the particles consist of quartz, chalcedony, or opal requires an accurate determination of either the index of refraction or the specific gravity, or both. The diagnostic criteria for opal have been given. It is suggested that the criterion for distinguishing fine-grained microfibrous quartz from chalcedony be based on the consideration that the index of refraction of quartz is always greater than 1.54 and that of chalcedony is always less than 1.54. This determination must in some cases be made with a high degree of accuracy, since some specimens of chalcedony have indices of refraction which approach 1.539.

Tridymite:

Tridymite is a mineral formed during the solidification of igneous rocks such as rhyolite and andesite. It is a relatively common mineral in such rocks, but it is never very abundant. It is a high-temperature modification of silica and has properties that are quite different from those of quartz. It occurs in crystals which belong to the orthorhombic system, and its principal indices of refraction are 1.469 and 1.473.

Acid and Intermediate Volcanic Glass:

Volcanic glass occurs in all varieties of volcanic rocks, and its presence is merely an indication that the cooling of the rock was sufficiently rapid so that complete crystallization did not take place. Materials such as obsidian and pumice represent volcanic rocks which solidified with such rapidity as to consist entirely of volcanic glass. Igneous rocks are described as "acid" if they contain more than 65 per cent silica, "intermediate" if they contain 55 to 65 per cent silica, and "basic" if they contain less than 55 per cent silica. Since volcanic glass may represent igneous rock material of each of these groups in the uncrystallized state, the glasses are referred to as acid, intermediate or basic as their silica content varies within these limits. Since the volcanic glass most frequently encountered in concrete aggregate materials occurs as interstitial filling around crystals in incompletely crystallized volcanic rocks, it is usually not practical to pick out samples of it for chemical analysis Chemical analysis is also a much more elaborate, expensive, and time-consuming process than is determination of index of refraction. It has been determined (8, 9) that acid and intermediate volcanic glass, that is to say volcanic glass with more than 55 per cent silica, has an index of refraction below 1.57 while basic volcanic glass has an index of refraction greater than 1.57. It is, therefore, possible to determine whether or not acid or intermediate volcanic glass is present in an igneous rock by preparing a powder from a fragment, embedding the powder in an immersion medium of index 1.57, and examining the mount using a petrographic microscope. George (8) has determined that the silica content of a natural glass can be estimated with an average error of but 3.4 per cent from determinations of index of refraction by the immersion method.

A new method for determining index of refraction has recently been described

(10) by Dodge which may prove to be of considerable usefulness in determinations of the sort just described. This method involves dark-field illumination and causes particles of differing index of refraction from the immersion medium to appear in various colors as a function of the degree of difference in index. This method does not involve the use of polarized light nor does it require the manipulation of the microscope for a determination on each particle. It has been used successfully (11) to distinguish particles of quartz from particles of chalcedony.

In summary, it is suggested that the techniques of petrography may be used to determine the presence or absence of any of the known reactive materials in concrete aggregates, to distinguish chalcedony from fine-grained quartz, and to distinguish acid or intermediate volcanic glass from basic glass. It is believed that studies using such techniques should, whenever possible, be made on aggregate samples which are subjected to chemical tests or performance tests for alkali-reactivity. Such studies will provide both a check on the results obtained by these other tests and also, in cases where reactivity is detected, will provide information on the nature of the material which is responsible for the reaction. One such correlated petrographic and performance-test research program has been reported in *Bulletin 229* of the Council for Scientific and Industrial Research of Australia (12). I should like to quote the last few sentences from this paper: "Support is given to published evidence that the presence of opal in aggregates must be regarded as dangerous and that the use of acid and intermediate volcanic rocks must be regarded with suspicion.... In several rocks cryptocrystalline quartz appeared to cause a dangerous reaction. Correlation of the behavior of mortars with the petrographic character of their aggregates suggests that much can be learnt from petrographic study. In many cases this should be sufficient to evaluate an aggregate. Although the use of low-alkali cement with a reactive aggregate may modify or delay deterioration, the evidence tends to show that such aggregates are undesirable for use with any cement."

REFERENCES

(1) Howard A. Coombs, "Expansion of Concrete Due to Reaction Between Andesitic Aggregate and Cement," *American Journal of Science*, Vol. 240, April, 1942, pp. 288–297.

(2) Thomas E. Stanton, discussion of a paper on "Studies Relating to the Mechanism by Which the Alkali-Aggregate Reaction Produces Expansion in Concrete," by W. C. Hansen, *Journal*, Am. Concrete Inst., June, 1944; *Proceedings*, Vol. 40, p. 228–3.

(3) Duncan McConnell, Richard C. Mielenz, William Y. Holland, and Kenneth T. Greene, "Cement-Aggregate Reaction in Concrete," *Journal*, Am. Concrete Inst., October, 1947; *Proceedings*, Vol. 44, pp. 93–128.

(4) Richard C. Mielenz, "Petrographic Examination of Concrete Aggregates," *Bulletin*, Geological Soc. Am., Vol. 57, April, 1946, pp. 309–318.

(5) Austin F. Rogers, "Blue Agate at Lead Pipe Springs, San Bernardino County, California," Abstract, *Bulletin*, Geological Soc. Am., December, 1947. (Paper presented April, 1947.)

(6) Bryant Mather, Discussion of a paper "Reactivity of Aggregate Constituents in Alkaline Solutions," by Leonard Bean and J. J. Tregoning, *Journal*, Am. Concrete Inst., November, 1945; *Proceedings*, Vol. 41, pp. 52–1 to 52–4.

(7) Williard H. Parsons, personal communication, March 9, 1948.

(8) William O. George, "The Relation of the Physical Properties of Natural Glasses to their Chemical Composition," *Journal of Geology*, Vol. XXXII, July–August, 1924, pp. 353–372.

(9) H. Buttgenbach, "Introduction à L' Étude des Roches Ignées," Paris and Liege, p. 49 (1938.)

(10) Nelson B. Dodge, "The Dark-Field Color Immersion Method," *American Mineralogist,* Vol. 37, September-October, 1948 pp. 541–549; Abstract, *Bulletin,* Geological Soc. Am., December, 1947.

(11) Nelson B. Dodge, personal communications, March 31 and April 15, 1948.

(12) A. R. Alderman, A. J. Gaskin, R. H. Jones, and H. E. Vivian, "Australian Aggregates and Cements," Part I of "Studies in Cement-Aggregate Reaction," *Bulletin No. 229,* Commonwealth of Australia, Council for Scientific and Industrial Research, pp. 7–46 (1947).

DISCUSSION

MR. HAROLD S. SWEET.[1]—I should like to ask Mr. Mather whether the fibrous character of micro-crystalline quartz seen in thin sections is sufficient to identify it as chalcedony, and conversely, if the lack of that fibrous nature is sufficient to call it something else?

MR. BRYANT MATHER (*author*).—I think not. We have many examples of materials which have been identified as chalcedony, which do not show any fibrous structure at the magnifications which we use. We have found examples of fibrous silica which show indices of 1.54 or more and therefore must be regarded as quartz. Work done by Donnay[2] and others on the structure of chalcedony has suggested that the fibers composing chalcedony may have diameters of the order of the wave length of light, and therefore in certain cases, if that is correct, you will not be able to observe the fibrous nature of the material.

MR. SWEET.—One other question: It is mentioned that the specific gravity or density of the material is indicative of the presence of opal or some other material. What typical values of the density or specific gravity of chalcedony could you give?

MR. MATHER.—They would be less than that of quartz. Chalcedony usually has an opal content of less than 15 per cent. However since the specific gravity of opal itself varies as a function of its water content that of chalcedony varies both as a function of the opal content and as a function of the water content of the opal. Reported values for the density of chalcedony range from 2.55 to 2.63.

MR. D. O. WOOLF.[3]—I should like to ask Mr. Mather whether he can tell us how the water in opal is held? In what state is it?

MR. MATHER.—A discussion of that could lead into complicated physical chemistry that I don't know a thing about. Opal is regarded as an amorphous semidehydrated gel. Therefore, while it is probably not the answer to your question, I can only say that the water in opal is regarded as held in the same way as water in any other gel. The physical chemistry of that is beyond me.

MR. HERBERT INSLEY[4] (*by letter*).—This important paper of Mr. Mather's not only provides a valuable list of the characteristic optical properties of some aggregate constituents known or suspected to be reactive with cement alkalies, but it also indirectly brings into focus two important problems, the answers to which are not now available and are required before intelligent specifications can be drawn on alkali limitations in cement or reactive constituents in aggregate. These are (1) the constitutional and compositional properties of aggregates which make them reactive, and (2) the steps which intervene between the primary chemical reaction and the physical expansion of the concrete.

---

[1] Research Engineer, Joint Highway Research Project with State Highway Commission of Indiana, Engineering Experiment Station, Purdue University, Lafayette, Ind.

[2] J. D. H. Donnay "La Biréfringence de Forme dans la Calcédoine," Ann. Soc. Géol. de Belg. Vol. LIX, pp. 289–302, Liége (1936).

[3] Senior Materials Engineer, Public Roads Administration, Federal Works Agency, Washington, D. C.

[4] Chief, Mineral Products Div., National Bureau of Standards, Washington, D. C.

Mr. Mather's reference to the definition of chalcedony as a mixture of opal and micro-fibrous quartz points up the first question. Since chalcedony is such a mixture, it is a rock and not a mineral. What are the reactive constituents of this mixture? Obviously opal is one. Is micro-fibrous quartz also reactive? If so, perhaps rocks containing micro-fibrous quartz without opal are also reactive? If only the opal is reactive, is any chalcedony dangerous no matter what the content of opal? It appears to be generally admitted that highly siliceous materials which are thermodynamically metastable at ordinary temperatures—opal, tridymite, acid and intermediate volcanic glasses—are reactive. Is the deleterious action of such materials dependent on the solubility in alkaline solutions, or is it dependent on the rate of solution? Specifically, may a very fine-grained quartz be reactive as contrasted with a coarse-grained quartz? It has been pointed out that some of the less silicic volcanic glasses are not reactive. Where is the dividing line and what are the refractive indices of glasses at this boundary?

The paper under discussion is less directly concerned with the second problem mentioned above, except as all petrographic work on concrete is concerned with it. Nevertheless, a satisfactory answer would remove the guess work connected with the prevention and cure of the alkali-aggregate problem. For example, pozzolanic material which is itself reactive with alkali is being added to cement to combat the expansion in the concrete resulting from the alkali-aggregate reaction. No completely satisfactory explanation for this behavior has been proposed. If this results from a more uniform distribution of osmotic cells, then the limiting grain size which promotes this action should be discovered.

Engineering organizations which are engaged in the erection of large monolithic concrete structures must, of necessity, devise specifications on limitations of alkalies and of aggregates for immediate use, and such specifications have to be based at present on cut-and-try data. The continued refinement of the specification limitation based on the same kind of data is expensive and wasteful. A more fundamental approach in investigation is needed in order to draw up intelligent specifications.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ON MINERAL AGGREGATES

The Symposium on Mineral Aggregates, sponsord by Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials, consists of a group of papers dealing with the distribution characteristics and uses of mineral aggregates. The papers were written primarily for those individuals who are interested in the characteristics and uses of mineral aggregates but who normally are not intimately connected with the aspects of research and testing. The following papers were presented and discussed at the Eleventh Session of the Fifty-first Annual Meeting of the American Society for Testing Materials held in Detroit, Mich., on June 24, 1948:

Distribution of Mineral Aggregates—K. B. Woods
Petrographic and Mineralogic Characteristics of Aggregates—Roger Rhoades and R. C. Mielenz
Physical and Chemical Tests of Mineral Aggregates and Their Significance—H. S. Sweet
Sampling of Mineral Aggregates—C. E. Proudley
Production and Manufacture of Fine and Coarse Aggregates—N. C. Rockwood
The Grading of Aggregates for Bituminous Construction—J. R. Benson
Grading of Mineral Aggregates for Portland-Cement Concrete and Mortars—W. H. Price
Influence of Mineral Aggregates on the Strength and Durability of Concrete—C. W. Allen
Lightweight Aggregates—R. E. Davis and J. W. Kelly
Mineral Aggregates for Bituminous Construction—J. T. Pauls and C. A. Carpenter
Mineral Aggregates for Low-Cost Roads and Water-Bound Macadams—E. A. Willis and J. A. Kelley, Jr.
Mineral Aggregates for Railroad Ballast—A. T. Goldbeck
Mineral Aggregates in the Chemical and Processing Industries and in Certain Other Uses—H. F. Kriege
Needed Research—D. O. Woolf

These papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 83, entitled, "Symposium on Mineral Aggregates."

SYMPOSIUM ON SPEED OF TESTING

INTRODUCTION

By P. G. McVetty[1]

This symposium is sponsored by the Section on Effect of Speed of Testing, Technical Committee on Mechanical Testing of Committee E-1. It follows a round-table discussion on speed of testing held at the Annual Meeting of the Society in 1947. Its purpose is to stimulate further study and improvement of the speed requirements in A.S.T.M. specifications.

Tests of some materials are not greatly affected by a considerable variation in testing speed. In others, the acceptance or rejection of a lot of material may depend upon the speed at which acceptance tests are conducted. In one case unnecessary speed restrictions in the specifications would increase the cost of the tests; in the other, lack of suitable speed limitations may result in rejections, delays, and increased cost.

The speed which affects test results is the rate of movement within the test specimen, but this is not easily measured or controlled without special equipment. In the region in which stress and strain are proportional, the more convenient rate of stressing or loading may be substituted for rate of straining. In many cases the rate of crosshead travel may be used, but it is essential to distinguish between rates measured under load and those measured without load.

A survey of A.S.T.M. specifications suggests the desirability of reviewing their speed requirements and clarifying the testing procedures involved. The papers included in this symposium discuss these factors for a wide variety of materials and indicate the limitations required and methods of measuring and controlling speeds.

---

[1] Manager, Strength of Materials Section, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa. Chairman of Symposium Committee.

TESTING SPEED LIMITATIONS FOR COMMITTEE A-1 SPECIFICATIONS FOR STEEL*

BY LAWFORD H. FRY[1]

In this section of the symposium the effect of speed in tension testing is considered in its relation to the acceptance specifications of Committee A-1 on Steel. The purpose of these specifications should be clearly understood. They do not serve to explore the hidden properties of materials in order to inform an inexperienced designer as to the uses to which a given material may be put. Metallurgical research may work to this end, but the acceptance specifications aim at another usefulness. It may be humbler but it is highly important.

An engineer designs a structure using certain materials and finds that it is satisfactory. He then wants to put it into production and to obtain a continuing supply of materials like those which have given good results. The steel specifications have been provided by Committee A-1 to control the supply of the steel required, to ensure as far as is practicable that the type and quality of the steel supplied shall remain uniform.

The primary responsibility of the specifications is to ensure a flow of uniform material, but their secondary responsibility is to see that control of this uniformity is effected without undue wear and tear on the nerves of the supplier. This concern for the supplier is not altruism on the part of the user. It is based on a realistic understanding of the fact that complexity in testing produces an increase in cost which by the immutable laws of economics is borne by the purchaser. Test requirements which serve no useful purpose waste time and increase the cost of manufacture to no good end.

Tension testing is one of the requirements put into the specifications in the attempt to secure uniform material. If uniform results are to represent uniform material, the conditions under which the results are obtained must be kept uniform.

One of the conditions which affects the results obtained is the speed at which the specimen is strained. Speed of testing must therefore be controlled and the problem of the writer of specifications is to determine the extent of the control required and to decide how the control can be applied most conveniently.

The effect of speed on the values found in the tension testing of steel has been studied extensively and a considerable body of information is available. It is well established that the values found for yield point, and to a smaller degree for tensile strength depend on the rate at which the specimen is strained. Consequently when tensile properties are specified, the speed of testing should be fixed. Fortunately, for our specifications the allowable range of speed is quite wide.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Mt. Vernon, N. Y. Deceased July, 1948.

Moore and Jones[2] reported on a large number of tests at widely varying rates of straining. They found for tensile strength and for yield strength that an increase of 800 per cent in the rate of straining had no serious effect on the values.

The author[3] reported to Subcommittee XIII of Committee A-1 on more than 300 tension tests. These tests showed that with steel with about 45,000 psi. yield point, the increase in yield point value produced by a threefold increase in rate of straining was approximately 3000 psi. which is of about the same order as the scatter between values obtained from a number of specimens of the same material at the same speed.

The figures given above are based on speeds obtainable with conventional tension testing machines, but for the sake of completeness, mention may be made of some really high-speed tests reported by Sir Geoffrey Taylor.[4] These tests made under ballistic conditions with rates of strain up to 10,000 in. per inch per second show that the dynamic value of the 0.2 per cent yield strength was about three times that of the static value for mild steel. With high tensile strength steels increase in yield strength was less.

Specification Requirements for Speed of Testing:

From the foregoing it is evident that for the A-1 acceptance specifications to be complete, they should provide limits for the rate of straining to be allowed in making tension tests. However in specifying the limits for the speeds to be used, the figures which have been quoted show that no high degree of precision is needed.

In the past when the majority of machines used in acceptance testing were of the screw driven type, it was convenient to control speed by limiting the free running crosshead speed. This is not theoretically correct because the tensile values depend on the rate of straining and for a given free crosshead speed different machines may give considerable variations in rates of straining. In practice however control by free crosshead speed is generally satisfactory, because the rate of straining need not be controlled to a high degree of precision.

Today the picture has been changed by the introduction of testing machines of modern design, particularly those with a pacing dial. As a result there is a tendency to change the practice and to specify a maximum for the loaded crosshead speed.[5]

The American Iron and Steel Institute in its pamphlet "Mechanical Testing Procedures and Definition for Plates and Sections" specifies for plates and structural shapes that any convenient speed may be used until the load reaches one-half of the value of the yield point and that beyond that the loaded crosshead speed shall not exceed $\frac{1}{16}$ in. per inch of gage length per minute until the yield point is reached. For determination of the ultimate strength the speed under load is not to exceed $\frac{1}{2}$ in. per inch of gage length per minute.

These requirements will undoubtedly produce satisfactory results. As between various machines there may be considerable variation in rates of straining for the same loaded crosshead speed, but with the crosshead speeds specified, such variations will not have any important effect on the uniformity of the results. If the loaded crosshead speed is to be used as a limiting factor with a screw driven machine, calibration of the machine should be made in advance of testing. Determination of crosshead speed during a test will not be convenient.

---

[2] P. G. Jones and H. F. Moore, "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 610 (1940).

[3] L. H. Fry, "Speed in Tension Testing and Its Influence on Yield Point Values," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 625 (1940).

[4] Geoffrey Taylor, "The Testing of Materials at High Rates of Loading," *Journal*, Institution of Civil Engrs., Vol. 26, No. 8, October, 1946.

[5] Crosshead speed is to be understood to mean the relative rate of separation of the two heads of the machine.

In the case of hydraulic machines with pacing dials, it may be more convenient to limit the rate of straining by limiting the rate of loading. The connection between rates of loading and straining is of course direct until the proportional limit is passed.

CONCLUSION

In drawing the steel acceptance specifications of Committee A-1, it must be recognized that the tensile properties to be obtained depend on the rate of straining applied in testing. A considerable variation in rate of strain can be allowed without undue variation in tensile properties being produced, but for the sake of completeness, some testing speed limits should be set. These should be laid down at the same time that tensile values are set, and for this the committee preparing the specification should be responsible.

Speed limits may be set by specifying limiting rates of crosshead speed under load or by specifying limiting rates of load application.

THE EFFECT OF SPEED OF TESTING ON MAGNESIUM-BASE ALLOYS*

By A. A. Moore[1]

Synopsis

The results of an extensive series of tension and compression tests at various speeds on magnesium-base alloys are reported. The yield strength was lowered by very low rates of straining where creep became a factor. At higher rates, approaching the maximum speed of the autographic recorder, no appreciable effect on the yield strength was obtained. The tensile strength and percentage of elongation were affected little by a considerable change of speed in the range normally used for tension testing.

Different means of specifying speed of testing are discussed.

As the art of testing has developed, new equipment and techniques have allowed the use of many refinements. Whether these refinements are necessary or even worthwhile must be decided on the basis of results obtained. The many factors which make up a test must be controlled to such an extent that universal duplication of results is achieved. One factor that has been under investigation for several years is the effect that speed of testing has on mechanical properties.

This paper reports the results of a considerable number of tension and compression tests on magnesium-base alloys at various speeds of testing. Several methods of determining the speed of testing were investigated.

Materials

In this paper, investigation of the effect of speed of testing is confined to magnesium-base alloys. Various conditions and heat treatments were selected to obtain a wide range of test properties. M1 sheet was tested in the annealed condition and AZ31X in the annealed and hard-rolled state. Extrusions used were 0.750 in. in diameter except one batch of M1 alloy tubing 0.750 in. in diameter with 0.032-in. wall thickness. The alloys tested as extrusions were AZ61X, M1, and AZ80X in the heat-treated and aged condition. Sand castings used were AZ63 alloy, solution heat treated and AZ92 and AZ63 alloys solution heat treated and aged. Nominal compositions and tensile properties are given in Table I. All materials were sampled for uniformity to help eliminate material variations.

Test Methods

Standard A.S.T.M. specimens were used with the usual type of wedge grips. Sheet specimens were 0.500 in. wide with a 2-in. gage length. The $\frac{3}{8}$-in. diameter extrusions in Table V had a 0.250-in. reduced section with a 2-in. gage length. The tubing was 0.750 in. in outside diameter with 0.032-in. wall thickness.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Division of Tests, Magnesium Laboratories, The Dow Chemical Co., Midland, Mich.

TABLE I.—COMPOSITION AND TENSILE PROPERTIES OF MAGNESIUM-BASE ALLOYS.

A.S.T.M. Alloy	Condition	Nominal Composition, per cent				Tensile Properties					
						Ultimate Strength, psi.		Yield Strength, psi.		Elong. in 2 in., per cent	
		Al	Mn	Zn	Mg	Typical	Spec. Min.	Typical	Spec. Min.	Typ.	Spec. Min.
AZ92	Sand Cast—HTA	9.0	0.1	2.0	Balance	40 000	34 000	23 000	18 000	2	1
AZ63	Sand Cast—HT	6.0	0.2	3.0	"	40 000	34 000	14 000	10 000	12	7
AZ63	Sand Cast—HTA					40 000	34 000	19 000	16 000	5	3
AZ31X	Annealed Sheet	3.0	0.3	1.0	"	37 000	32 000	22 000	...	21	12
AZ31X	Hard Rolled Sheet					43 000	38 000	33 000	26 000	11	4
AZ61X	Extruded Rod	6.5	0.2	1.0	"	45 000	40 000	32 000	26 000	15	11
M1	Extruded Rod	...	1.5	...	"	38 000	30 000	26 000	20 000	10	3
M1	Extruded Tubing					33 000	28 000	21 000	...	9	3
M1	Annealed Sheet					33 000	28 000	15 000	...	17	12
AZ80X	Extruded Rod—HTA	8.5	0.2	0.5	"	51 000	47 000	35 000	30 000	9	5
AZ91	Die Cast	9.0	0.2	0.6	"	33 000	...	22 000	...	3	...

About 4½ in. of tubing was not supported by plugs. Standard 0.505-in. diameter round bars were used on the extruded rod and castings.

The tests were made on three hydraulic testing machines. One had a capsule support connected to a Bourdon tube in the weighing system. The second machine used a lapped ram in connection with a Bourdon tube weighing system. The third machine had a lapped ram connected to a rotating cylinder with a lapped ram and pendulum for a weighing system.

The time to the yield strength was taken with a stop watch to the point where the extensometer was removed. The graph generally was taken a little beyond the yield strength. The time from the yield strength to rupture was also obtained with a stop watch. The strain to the yield strength was taken from the autographic record. The strain from yield strength to rupture was taken as the difference between the strain from the graph and the percentage of elongation after rupture. A dial gage on the head of the testing machine indicated the crosshead movement under load.

The rate of stressing and rate of straining are given as average values. No strain or load pacer was used to obtain uniform strain or stress application.

TABLE II.—EFFECT OF SPEED OF TESTING ON TENSILE YIELD STRENGTH OF MAGNESIUM-BASE ALLOYS.

A.S.T.M. Alloy	Condition	Yield Strength, psi.		
		Crosshead Speed, in. per min.		
		0.02	0.05	0.10
AZ61X	Extrusion	33 400	33 600	33 600
M1	Extrusion	29 000	29 000	28 100
M1	Extrusion (Tubing)	15 500	15 600	15 200
AZ80X	Extrusion—HTA	41 300	42 200	42 700
M1	Annealed Sheet—0.091 in. thick	18 900	19 200	19 300
M1	Annealed Sheet—0.125 in. thick	17 100	19 600	19 300
AZ31X	Annealed Sheet—0.091 in. thick	21 700	21 700	21 800
AZ31X	Annealed Sheet—0.125 in. thick	22 900	23 100	23 100
AZ31X	Hard Rolled Sheet—0.091 in. thick	33 300	33 000	32 700
AZ31X	Hard Rolled Sheet—0.125 in. thick	33 000	33 100	33 300

The rates of straining to the yield strength were selected to cover a range wider than would normally be used. The fastest speed was just under the capacity of the recorder system. The slowest speed was about one fifth as fast. From the yield strength to rupture, the ratio of the fastest to the slowest speed was a hundred to one.

The compressive yield strength values given in Table VI were obtained on the two sizes of round specimens indicated. Strain was measured between the mandrels autographically. The crosshead travel would be nearly the same as the strain in the specimen.

The stress values given in the tables are a combination of the reaction of the machine to the speed used as well as the effect on the material. All machines were calibrated according to A.S.T.M. Tentative Methods of Verification of Testing Machines (E 4 - 47 T).[2] It is known from the work done by the National Bureau of Standards[3] that errors of load indication may be encountered with increasing rates of load application.

TEST DATA AND DISCUSSION

The tests reported in the paper were made at several different times with different purposes in view, so the data presented may seem somewhat disorganized. Older specifications on screw-type testing machines were based on the free running crosshead speeds which were a reasonable approximation of the crosshead speed under load.

TABLE III.—EFFECT OF SPEED OF TESTING ON TENSILE STRENGTH AND ELONGATION OF MAGNESIUM-BASE ALLOYS.[a]

Crosshead Speed, in. per min.		0.02		0.05		0.2		0.5		1.0		1.5		2.0	
A.S.T.M. Alloy	Condition	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent
Z61X......	Extrusion	47 100	15.8	47 100	15.8	46 600	15.5	46 400	15.0	46 200	15.2	46 100	15.0	45 800	14.5
M1........	Extrusion	34 300	12.5	35 800	15.3	37 800	16.0	37 700	13.5	37 900	10.2	38 400	10.0	38 800	9.5
M1........	Extrusion (tubing)[b]			28 900	25.1	30 200	21.6	31 000	20.0	31 500	19.2	31 700	18.4	31 600	16.0
AZ80X....	Extrusion—HT	55 400	8.0	55 900	7.1	56 100	5.8	56 200	5.3	56 800	5.1	56 300	4.7	56 600	4.4
M1.......	Annealed Sheet, 0.064 in. thick	29 800	24.0	31 600	20.2	33 200	17.0	33 300	16.7	34 200	16.2	33 700	15.5	34 500	14.8
M1.......	Annealed Sheet, 0.091 in. thick	29 800	21.7	31 000	19.8	32 500	16.8	33 300	17.0	32 700	16.5	33 100	15.5	33 300	15.3
M1.......	Annealed sheet, 0.125 in. thick	29 800	24.2	30 800	23.5	32 200	21.2	32 600	19.7	33 400	18.2	33 600	17.7	33 900	16.8
AZ31X....	Annealed sheet, 0.064 in. thick	36 700	28.0	37 600	24.3	37 800	23.2	37 500	22.0	37 100	21.0	36 800	21.3	36 900	20.7
AZ31X....	Annealed sheet, 0.091 in. thick	36 700	27.0	36 700	25.7	36 800	23.7	36 700	23.0	35 900	22.3	35 700	21.5	35 600	21.3
AZ31X....	Annealed sheet, 0.125 in. thick	36 300	26.7	36 500	23.8	36 800	20.2	36 900	22.5	36 500	20.8	36 400	20.5	36 500	19.7
AZ31X....	Hardrolled sheet, 0.064 in. thick	43 200	14.7	44 100	13.3	44 300	10.5	44 200	10.0	43 400	9.5	43 900	9.7	44 000	8.7
AZ31X..	Hard rolled sheet, 0.091 in. thick	41 700	13.0	42 600	13.5	43 000	10.3	42 800	10.8	42 000	9.7	42 100	8.2	42 000	8.0
AZ31X..	Hard rolled sheet, 0.125 in. thick	42 200	20.5	42 000	17.5	42 500	14.0	42 500	12.3	42 300	10.2	42 300	12.2	42 200	12.2
AZ63......	Sand casting—HT									43 100	17.0	42 300	15.9	42 500	15.3
AZ63......	Sand casting—HTA									43 800	6.8	43 900	6.0	42 200	5.5
AZ92......	Sand casting—HTA									39 100	1.3	42 100	1.2	40 200	1.2

[a] All values are the average of three or more tests. Wedge grips were used in all tests.
[b] Tubing size 0.750 in. O.D. by 0.032-in. wall thickness. Approximately 4.5 in. of test section between plugs.

---

[2] 1947 Supplement to Book of A.S.T.M. Standards, Part I-A, p. 366; Part I-B, p. 307; Part II, p. 436; Part III-A, p. 420; Part III-B, p. 285.

[3] Bruce Wilson and Carl Johnson, "Calibration of Testing Machines Under Dynamic Loading," *Journal of Research*, Nat. Bureau Standards, Vol. 19 (1937).

TABLE IV.—EFFECT OF SPEED OF TESTING ON TENSILE PROPERTIES OF DIE-CAST MAGNESIUM.

Crosshead Speed to Yield Strength, in. per min.	Yield Strength, psi.	Time to Yield Strength, sec.	Average Rate of Stressing to Yield Strength, psi. per min.	Average Rate of Straining to Yield Strength, in per inch per min.	Ratio of Crosshead Movement to Strain in Specimen for 2-in. Gage Length to Yield Strength	Crosshead Speed from Yield Strength to Rupture, in. per min.	Tensile Strength, psi.	Elongation in 2 in., per cent	Time from Yield Strength to Rupture, sec.	Average Rate of Stressing from Yield Strength to Rupture, psi. per min.	Average Rate of Straining from Yield Strength to Rupture, in. per inch per min.	Ratio of Crosshead Movement to Strain in Specimen for 2-in. Gage Length from Yield Strength to Rupture
0.112	26 500	131	12 100	0.0046	12.2	0.20	34 800	2.2	21	23 700	0.057	1.75
0.224	26 300	74	21 300	0.0081	13.8	0.314	34 800	3.1	16	32 200	0.108	1.45
0.308	27 000	52	31 200	0.0115	13.3	0.980	35 700	3.0	11	42 000	0.162	1.52
0.985						0.910	34 800	2.5	7	70 000	0.188	2.30
1.315						1.50	35 000	2.9	4	126 000	0.405	1.85

NOTE.—Test values are the average of four die cast AZ91 alloy test bars. Wedge grips were used.

TABLE V.—RELATION OF CROSSHEAD MOVEMENT TO RATES OF STRESSING AND STRAINING IN MAGNESIUM-BASE ALLOYS.

Time to Yield Strength, sec.	Strain in 2-in. Gage Length to Yield Strength, in.	Crosshead Movement to Yield Strength, in.	Yield Strength, psi.	Crosshead Speed to Yield Strength, in. per min.	Average rate of Straining to Yield Strength, in. per inch per min.	Average Rate of Stressing to Yield Strength, psi. per min.	Time from Yield Strength to Rupture, sec.	Strain in 2-in. Gage Length Yield Strength to Rupture, in.	Crosshead Movement Yield Strength to Rupture, in.	Tensile Strength, psi.	Average Crosshead Speed Yield Strength to Rupture, in. per min.	Average Rate of Straining Yield Strength to Rupture, in. per inch per min.	Average Rate of Stressing Yield Strength to Rupture, psi. per min.	Ratio: Col. 3/Col. 2	Ratio: Col. 10/Col. 9
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	*14*	*15*	*16*
Extruded Strip 0.060 in. Thick															
62	0.021	0.125	20 300	0.121	0.010	19 700	20	0.339	0.470	38 400	1.41	0.51	115 000	6.0	1.4
34	0.017	0.100	18 500	0.176	0.015	32 600	16	0.273	0.380	32 400	1.34	0.48	114 000	5.9	1.4
42[a]	0.019	0.110	19 700	0.157	0.014	28 000	19	0.305	0.420	35 600	1.33	0.48	113 000	5.8	1.4
⅜-in. Diameter Extrusions															
68	0.022	0.168	30 600	0.148	0.0097	27 000	22	0.220	0.262	45 100	0.71	0.30	39 600	7.7	1.2
47	0.022	0.165	34 000	0.210	0.014	43 400	12	0.298	0.295	61 500	1.47	0.74	137 000	7.4	1.0
57[b]	0.022	0.183	35 100	0.193	0.012	36 900	11	0.216	0.219	49 700	1.19	0.59	79 700	8.4	1.0
¾-in. Diameter Bars from Castings															
59	0.031	0.258	19 700	0.262	0.013	20 000	24	0.096	0.192	35 500	0.48	0.12	38 700	8.3	2.0
35	0.024	0.180	19 400	0.308	0.020	33 200	24	0.088	0.180	35 600	0.45	0.11	40 500	7.5	2.05
48[c]	0.026	0.241	19 700	0.302	0.016	24 600	23	0.086	0.183	35 400	0.48	0.11	40 800	9.3	2.13

[a] Average of 24 test specimens.
[b] Average of 22 test specimens.
[c] Average of 4 test specimens.

One of the first steps taken in the attempt to evaluate the effect of speed on magnesium-base alloys was to determine a means of obtaining rates of straining that would be accurate. It was soon found that the free-running crosshead speed was useless on the hydraulic machines in general, although some were better than others. Ratios as high as 10 and 12 to 1 were found between the free-running speed and the rate of travel under load with the same valve setting.

The head speed under load was used as a means of determining rates of testing for the alloys given in Tables II and III. The main objective was to determine the effect of speed on properties in the range that might be used for commercial testing. The slow speeds were so slow that creep was a definite factor.

After determining the effect of the different rates, the problem of a satisfactory means of specifying the speed became evident. Any system should employ only the instruments commonly available in the laboratory. This work resulted in the values given in Tables IV and V.

The universal adoption of the autographic stress-strain recorder should tend to standardize rates of straining to the yield strength. As a matter of economy, the maximum speed obtainable is used. On the recorders used in this work, free-running drum speeds ranged from 11 to 12 in. per minute. With a magnification of 250 to 1, the maximum strain that could be recorded would be 0.048 in. per minute. With a 2-in. gage length this would be 0.024 in. per inch of gage length per minute. The fastest average rate of straining obtained was the 0.015 in. per inch per minute given in Table V. The minimum rate would be governed only by the desire of the operator.

The ratio of the head movement under load to the strain varied for different types of specimens. The die-cast bars given in Table IV gave a ratio of 12.2 to 13.8 regardless of the speed used. For other types of bars given in Table V, the ratio fell as low as 5.8. The maximum average rate of stressing obtained was 43,400 psi. per minute to the yield strength.

The rate of straining is preferred as an exact means of specification since the type of grips and other machine factors need not be taken into consideration. The crosshead travel is a reasonable approximation of the strain beyond the yield strength, as is shown in Tables IV and V where the ratio of crosshead movement to strain ranges from 1.0 to 2.3 with an average of about 1.5. For threaded or shouldered specimens, the ratio would be near 1.0. Rate of stressing cannot be used satisfactorily beyond the yield strength because the load may be decreasing beyond the maximum where necking occurs.

The results given in Table III and IV show little effect on the tensile strength and elongation of magnesium-base alloys in the range of speed normally used. At crosshead speeds of 0.02 and 0.05 in. per minute, the tensile strength is decreased and the percentage of elongation increased. At 0.2 in. per minute, which corresponds to a rate of strain of about 0.07 in. per minute, it would take 3 min. to complete a test with 21 per cent elongation. This is considered too slow for efficient operation. Crosshead speeds most commonly used are in excess of 0.5 in. per minute. In this range the values for magnesium remain fairly constant with the biggest difference being found in M1 alloy. The greatest change is found in the elongation values.

The tensile yield strength values show little change with a five-fold increase in the rate of crosshead travel. Since the strain is proportional to the crosshead travel for a given set of conditions, regardless of speed, the rate of straining would vary by a factor of five. The effect of speed on the compressive yield values given in Table VI are about the same as for the tensile yield values in Table II.

TABLE VI.—COMPRESSIVE YIELD STRENGTH OF MAGNESIUM-BASE ALLOYS AT DIFFERENT RATES OF STRAINING.

Alloy and Condition	Compressive Yield Strength, psi.	
	Crosshead Speed, 0.022 to 0.036 in. per min.	Crosshead Speed, 0.0025 to 0.005 in. per min.
Sand Cast AZ63— HT[a]	21 900	22 200
Sand Cast AZ63—HTA[a]	22 000	21 700
Extruded AZ61[b]	20 100	20 150
Extruded M1[b]	20 000	20 000
Extruded M1[b]	12 850	13 200
Extruded AZ80X—HTA[b]	39 700	39 600
Extruded AZ80X—HTA[b]	40 400	40 600

[a] Specimen size—⅜ in. diameter by 1⅛ in. long.
[b] Specimen size—½ in. diameter by 1½ in. long.

SUMMARY AND CONCLUSIONS

No special equipment is necessary to obtain a reasonable evaluation of the effect of the speed of testing. Average values must be used unless strain or stress pacers are available. Rate of stressing or rate of straining are equally satisfactory to the yield strength, since both may be obtained from the autographic record. From the yield strength to the breaking point, the average rate of straining may be obtained from the percentage elongation. The crosshead travel under load is a reasonable approximation of the strain between the yield strength and rupture.

The properties of magnesium-base alloys were affected but little by the changes in speed that would normally be found in commercial testing.

SPEED OF TESTING OF WOOD: FACTORS IN ITS CONTROL AND ITS EFFECT ON STRENGTH*

BY L. J. MARKWARDT AND J. A. LISKA[1]

SYNOPSIS

Early studies of the mechanical properties of wood showed that test results are influenced by rate of loading and that the ultimate strength is reduced with increased duration of stress. Accordingly, test methods for wood have consistently included limitations on speed of testing. Data are presented from recent studies on the effect of rate of loading on the compressive strength parallel to grain and on flexural strength.

The application of speed of test requirements, including restrictions on rate of strain or rate of stress, is dependent on the limitations and characteristics of the testing machine and related equipment. Data are presented from an analysis of hydraulic and mechanical drive testing machines indicating sources of error that may affect the speed of test. It is recognized that the loaded crosshead speed will be less than the free running crosshead speed, but it is less obvious that the loaded crosshead speed, as measured by a rate-of-strain pacer, may also be in error. Included also is a discussion of the problem of specifying speed of test. Suggestion for consideration is made of the possibility of limiting speed of test by simply requiring that the load be applied uniformly and continuously at such a rate that the maximum load will be reached in not less than some specified time, the time being dependent on the particular test, the test conditions, and the material.

Early investigators of the mechanical properties of wood were conscious of the effect of rate of loading on the strength of wood and the necessity for standardization of speed of testing requirements. W. K. Hatt (**3**),[2] in his manual "Instructions to Engineers of Timber Tests," published in 1906, presented certain fundamental ideas and requirements with respect to method of conducting timber tests that are still in effect. H. D. Tiemann (**5**) and McGarvey Cline (**2**) in 1908 published results of experiments to evaluate the effect of rate of application of load on the strength of wood members and proposed speed of testing requirements for several basic test procedures. More recently the need arose for more detailed and extensive data on the effect of rate of loading and duration of stress, involving several strength properties and a number of different species of wood. New techniques and methods were developed at the Forest Products Laboratory for obtaining information on the stress increase for loading times of a fraction of a second, as in aircraft, and for evaluating the behavior of structural members under long-time continuous or intermittent loading. The results of these various laboratory investigations together with data on the characteristics of a number of testing machines are presented here to provide a basis for consideration of the problem of establishing specification requirements for speed of testing.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Assistant Director and Engineer, respectively, Forest Products Laboratory, U. S. Department of Agriculture Madison, Wis.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1159.

PURPOSE OF INVESTIGATION

The studies undertaken at the laboratory during the past several years have been made with a twofold purpose in mind, first, to determine the actual rate of loaded crosshead movement or rate of strain application during test and, second, to investigate more fully the effect of rate-of-load application on the mechanical properties of wood, particularly in compression parallel to grain and flexure. All of the work reported herein has been carried out using standard testing machines and does not include shock loading or dead load tests. Work on the latter subject is in progress, however, as are studies of the effect of intermittent loading and repeated loading on strength.

OPERATING CHARACTERISTICS OF TESTING MACHINES

Methods of Specifying Speed of Test:

The speed of testing in "static" type testing machines of the mechanical or hydraulic loading type can be controlled by rate of strain application, rate of stress application, by a combination of the two, or simply by stating that the test shall be completed in a certain specified time. In each case the purpose of specifying a testing speed is to provide for uniform test results for materials affected by rate of load application. Obviously, the rate of loading should be such as to provide sufficient time to permit taking of any required load-deformation data. Furthermore, the control method should be as simple as possible, consistent with achieving the desired result, both from the standpoint of simplicity and clarity of the specification and of satisfactory utilization of a wide variety of testing machines.

It is evident that the rate-of-strain and rate-of-stress methods will give identical results to the proportional limit of an elastic material; but beyond that point, for all but brittle materials, a constant rate of stress application is difficult if not impossible to maintain, since the stress increases only slightly with a large increase in deformation. A combination of the two methods using a rate-of-strain pacer to control platen speed beyond the proportional limit could be adapted to most hydraulic machines equipped with pacers but would not be suitable for testing machines of the mechanical-drive type unless they were equipped with a pacing device and continuously variable speed control. It is believed that rate-of-strain control can be most universally applied, since all testing machines have some rate of crosshead movement control. An understanding of the variation of crosshead movement under load is necessary, however, if the principles underlying the speed requirements are to be followed.

That the loaded crosshead speed is less than the free-running head speed for a mechanical-type testing machine is generally recognized, but the limitations of some hydraulic-type testing machines are probably not fully appreciated. What may be even less apparent, moreover, to those who have not carefully examined the characteristics of testing machines, is the fact that the loaded crosshead speed for two mechanical-type machines or two hydraulic-type machines may be essentially different even though the free-running head speeds are the same. It is also true that the loaded crosshead speed of a testing machine will vary in testing specimens of the same essential strength but of different lengths or in testing specimens of the same length but of different strengths and elastic moduli even though the free-running head speed is the same. These differences are a function of the total stiffness of the test specimen and the over-all stiffness of the testing machine. This latter stiffness may be separated into elastic and nonelastic characteristics. In order to investigate these characteristics of testing machines more completely so that they could be given proper consideration in the establishment of speed-of-testing requirements and the manner in which these requirements could be best expressed to obtain the desired conformity in testing procedures and test results, a number of investigations on the variation in testing machine speeds were made.

Testing Machine Performance Studied:

In 1937, Groth and Freas made a study of the characteristics of one 30,000-lb. capacity and one 200,000-lb. capacity testing machine of the mechanical-drive type and one 100,000-lb. capacity testing machine of the hydraulic-drive type. The hydraulic machine had a lapped ram and a pacing disk attachment for controlling the rate of deformation or rate of movement of the loading head with respect to the base of the testing machine. This study was made in order to determine the causes of variation in testing machine speeds and the sources of error that affect the loaded crosshead speed. They reported no noticeable change in motor speed and no material belt slippage as the loaded crosshead speeds changed during operation of the two mechanical-type testing machines. They did show, however, that the loaded crosshead speed on these machines was influenced to a greater or less degree by the following factors:

(*a*) Stretch in screws,
(*b*) Play between screws and loading head,
(*c*) Deflection of loading head,
(*d*) Movement of weighing platform,
(*e*) Play between knife edges and plates of weighing platform,
(*f*) Play between thrust bearing and base, and
(*g*) Deflection of weighing platform.

The speed characteristics of the hydraulic-type testing machine were found to vary with the following:

(*a*) Stretch in screws,
(*b*) Play between screws and loading head,
(*c*) Deflection of loading head,
(*d*) Play between columns and stationary head, and
(*e*) Deflection of stationary head.

To these factors affecting loaded crosshead speed must be added the effect of temperature or viscosity of the oil in hydraulic machines and the type of hydraulic loading, whether through a lapped or packed ram. Another important factor affecting rate of loading is the slippage in nonelastic-type fittings, such as wedge-type grips.

Analysis of these factors indicates that the stretch in the screws and deflections in the loading and stationary heads are elastic types of deformation that are generally proportional to the applied load for any specified testing machine condition in both the mechanical- and hydraulic-type machines. The stretch in the screws will vary in accordance with the effective length of the screws in any test and with the size and number of screws. If the test specimen in a mechanical machine undergoes any considerable deformation during test, the loaded crosshead speed may change materially, since the effective length of the screws is changed.

The nonelastic deformations that cause play or movement between testing machine parts are more or less fixed

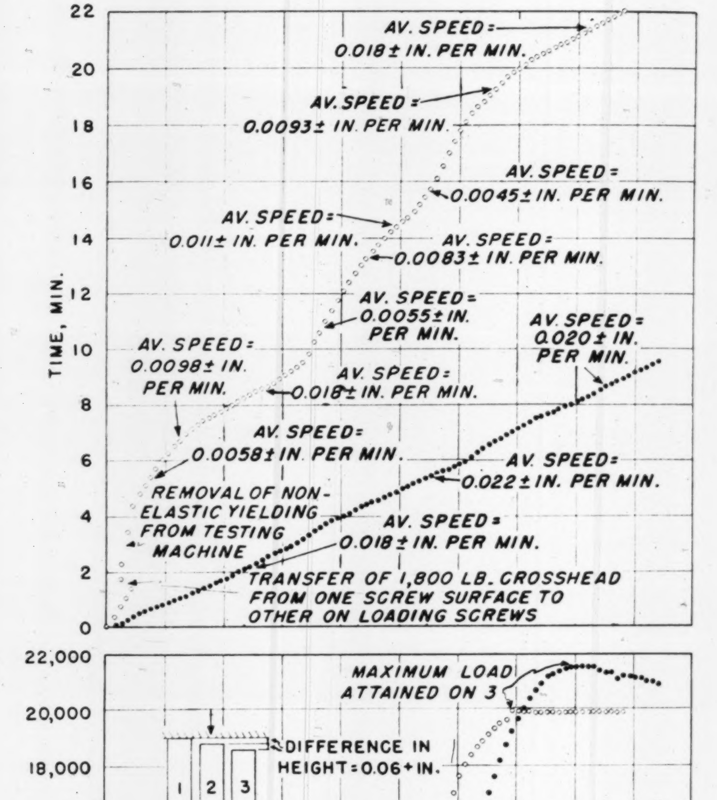


FIG. 1.—Comparison of Rate of Loading Characteristics of a Mechanical- and a Hydraulic-Type Testing Machine for Tests of Three 1 by 1 by 4-in. Sitka Spruce Compression Specimens.

errors that depend somewhat on the condition of the particular testing machine in question and normally affect crosshead speed in the low load ranges until all the play is taken out of the loading system. In testing a specimen in a machine with a screw-driven loading head, the weight of the head is first released on the specimen, and then some lost motion ensues as the bearing of the movable head is transferred from one surface to the other of the drive screw. An example of this type of deformation involving the transfer of an 1800-lb. loading head from one screw surface to another in a mechanical type machine may be noted in Fig. 1.

Behavior of Machines under Load:

Subsequent to these examinations for possible sources of error in loaded crosshead speed, these investigators made strength tests on compression parallel-to-grain test specimens having ultimate strengths of approximately 32,000 lb. and proportional limit strengths of about 24,000 lb. It was found that a large part of the loss in loaded crosshead speed in mechanical-type testing machines occurred at low loads, as shown in Table I, probably indicating that most of the error due to nonelastic deformation was taking place at this load level. This same characteristic was true for the hydraulic machine of the lapped-ram type, but to a somewhat lesser extent. An actual increase in speed of the loaded crosshead was noted for all machines after the proportional limit of the test specimens had been reached.

TABLE I.—CHARACTERISTIC VARIATIONS IN CROSSHEAD SPEED FOR THREE TESTING MACHINES.

Machine	Speed, in. per min.				Speed, per cent of no-load speed	
	At No Load	At 2000 lb.	At 24,000 lb.	Change	At 2000 lb.	At 24,000 lb.
100,000-lb. hydraulic.......	0.025	0.012	0.022	0.010	48.0	88.0
200,000-lb. mechanical.....	0.029	0.012	0.019	0.007	41.4	65.5
30,000-lb. mechanical.....	0.025	0.007	0.012	0.005	28.0	48.0

Measurements of the sources of error or factors that affect the loaded crosshead speed at a load of 24,000 lb. indicated that elastic deformation accounted for about 61 per cent of the error in the hydraulic-type testing machine, while about 39 per cent was due to nonelastic deformation. It was not possible to break down the error in this way for the mechanical-type machines, but it should be noted that stretch in the screws accounted for about 1 per cent of the error in the 200,000-lb. machine and about 3 per cent in the 30,000-lb. machine, showing the relative effect of the elastic stiffness of the screws.

In a subsequent study to evaluate further the characteristics of testing machines, Ernst, Empey, and Jones confirmed the observations of Groth and Freas that the actual loaded crosshead speed increased toward the end of the test. They did not attempt to segregate the possible sources of error in this investigation but rather attempted to evaluate the effect of specimen and machine characteristics on the speed of testing and to determine the effectiveness of a rate-of-strain pacer on the control of rate of head movement. Their studies were made on two mechanical-type testing machines of 10,000-lb. capacities, one mechanical-type machine of 25,000-lb. capacity and one 120,000-lb. capacity testing machine of the packed-ram hydraulic-drive type, complete with rate-of-strain pacer indicating the rate of movement of the loaded crosshead with respect to the base of the testing machine.

These investigations established the fact that the actual loaded crosshead speed was dependent on two factors (1) the total stiffness of the specimen, and (2) the total stiffness of the testing machine. The total stiffness of the specimen was defined as being equal to the increment in load in pounds divided by the increment in head travel in inches and was termed "load modulus" to differentiate it from modulus of elasticity. The stiffness of the machine is dependent on the elastic characteristics of the loading system and the effective length of the screws, it being assumed that the nonelastic yielding is overcome early in the test. After testing several specimens in each of the testing machines under consideration in this program and comparing the data obtained by taking simultaneous readings of load, head travel and time for each test, it was found in each case that the speed of the loaded crosshead was reduced as the load modulus was increased. The total stiffness of the testing machine remained essentially constant during these tests.

Special Load Head-Travel Time Study:

In order to demonstrate more completely the effect of load modulus on loaded crosshead speed as well as to determine the effectiveness of a rate-of-strain pacer in controlling the actual loaded crosshead speed a special test was performed. Three specimens 1 by 1 in. in cross-section and approximately 4-in. long were placed side by side on the head of a 25,000-lb. capacity mechanical-type testing machine, and a matched set was placed in a 120,000-lb. hydraulic testing machine of the packed ram type, and in each instance the group of specimens was tested in compression. The three specimens in a group differed in length from one another by graduated amounts estimated to be more than the deformation at maximum load but less than the amount that would cause complete failure. By testing these three specimens simultaneously, the load on the testing machine continued to increase, except for short periods of time, as each specimen reached maximum load. The load modulus decreased approximately to zero as each specimen reached maximum load but increased approximately to its initial value as the load was picked up by the next shorter specimen. In this way, if loaded crosshead speed is independent of load and varies with the load-modulus, the loaded crosshead speed should increase to approximately free running crosshead speed each time a maximum load is reached, irrespective of its magnitude. If the rate-of-strain pacer on the hydraulic machine affords adequate control of the crosshead movement, the crosshead speed should be the same throughout the test.

The data obtained in these tests by taking simultaneous readings of load, crosshead travel, and time are plotted in Fig. 1. The load – crosshead travel curves for both the mechanical- and hydraulic-type testing machines are shown in the lower portion of the figure with corresponding time – crosshead travel directly above. Average rates of loaded crosshead movement are indicated along the length of the speed-time curves and indicate in each instance the midpoint of a 1-min. time interval used in calculation of the average value.

The load-head travel curves are essentially equivalent for the tests made in the mechanical- and hydraulic-type testing machines. In each test, the normal characteristics of a load-deformation curve are noted up to the maximum load on the longest specimen. The deformation then increases without an increase in load until bearing on the next shorter specimen is completed, when the load and deformation again increase in an orthodox manner and so on until the test is completed.

The time – crosshead travel curves, however, are markedly different. In the case of the mechanical-type testing

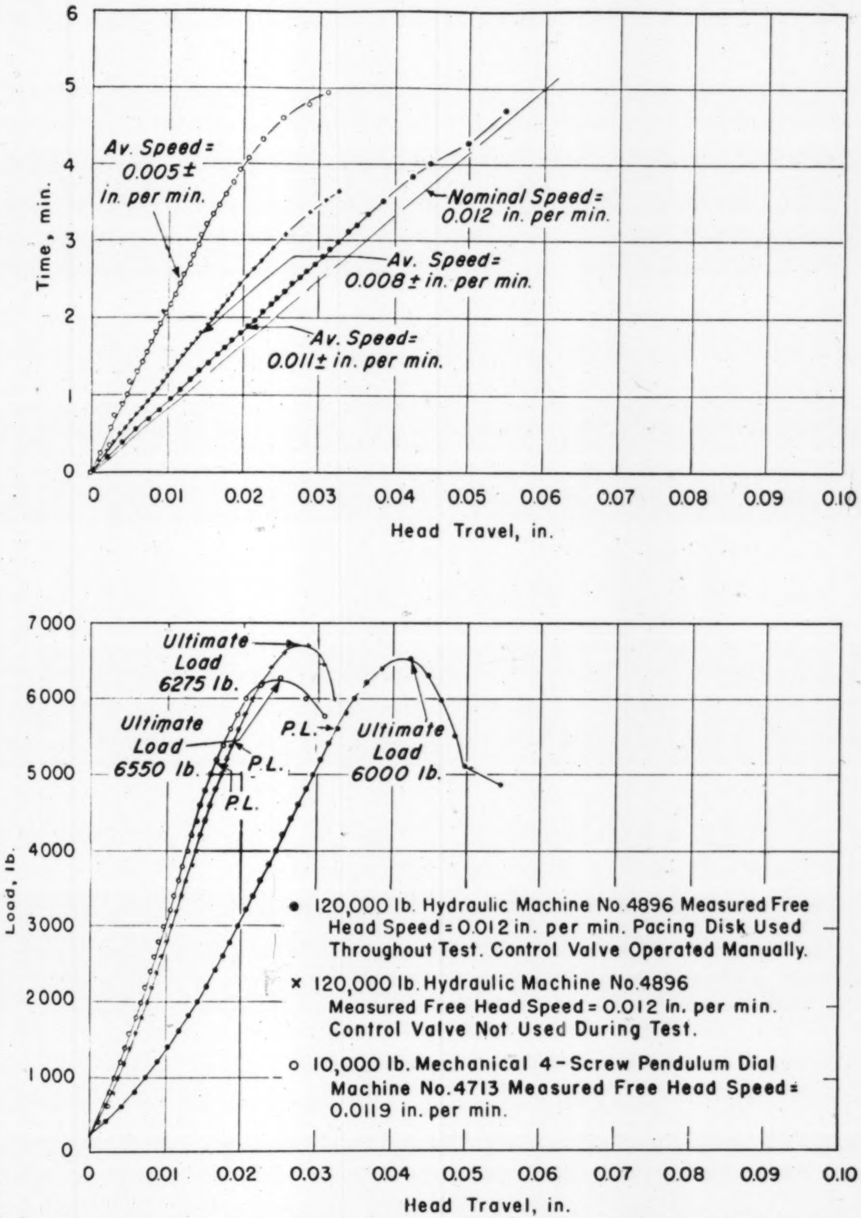


FIG. 2.—Comparison of the Rate of Loading Characteristics of a Mechanical- and a Hydraulic-Type Testing Machine for Tests of 1 by 1 by 4-in. Douglas-Fir Compression Specimens.

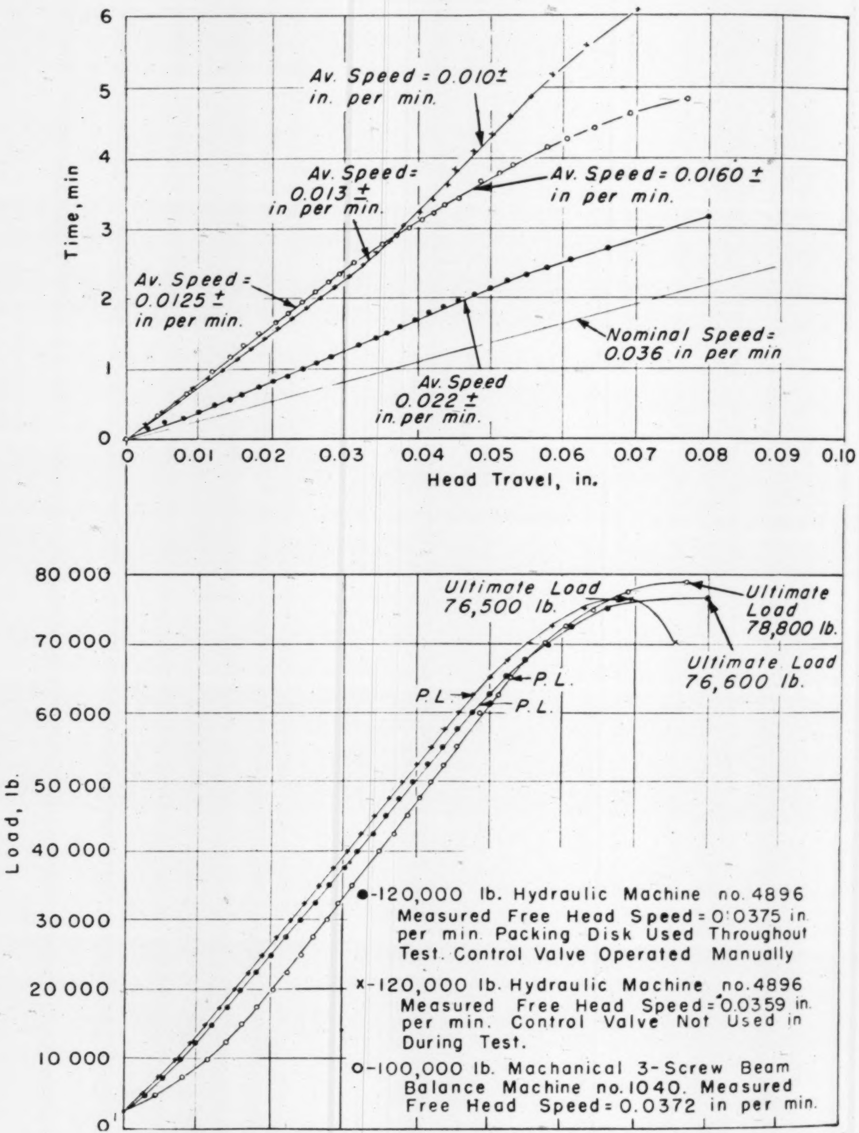


FIG. 3.—Comparison of the Rate of Loading Characteristics of a Mechanical- and a Hydraulic-Type Testing Machine for Tests of 3 by 4 by 12-in. Douglas-Fir Compression Specimens.

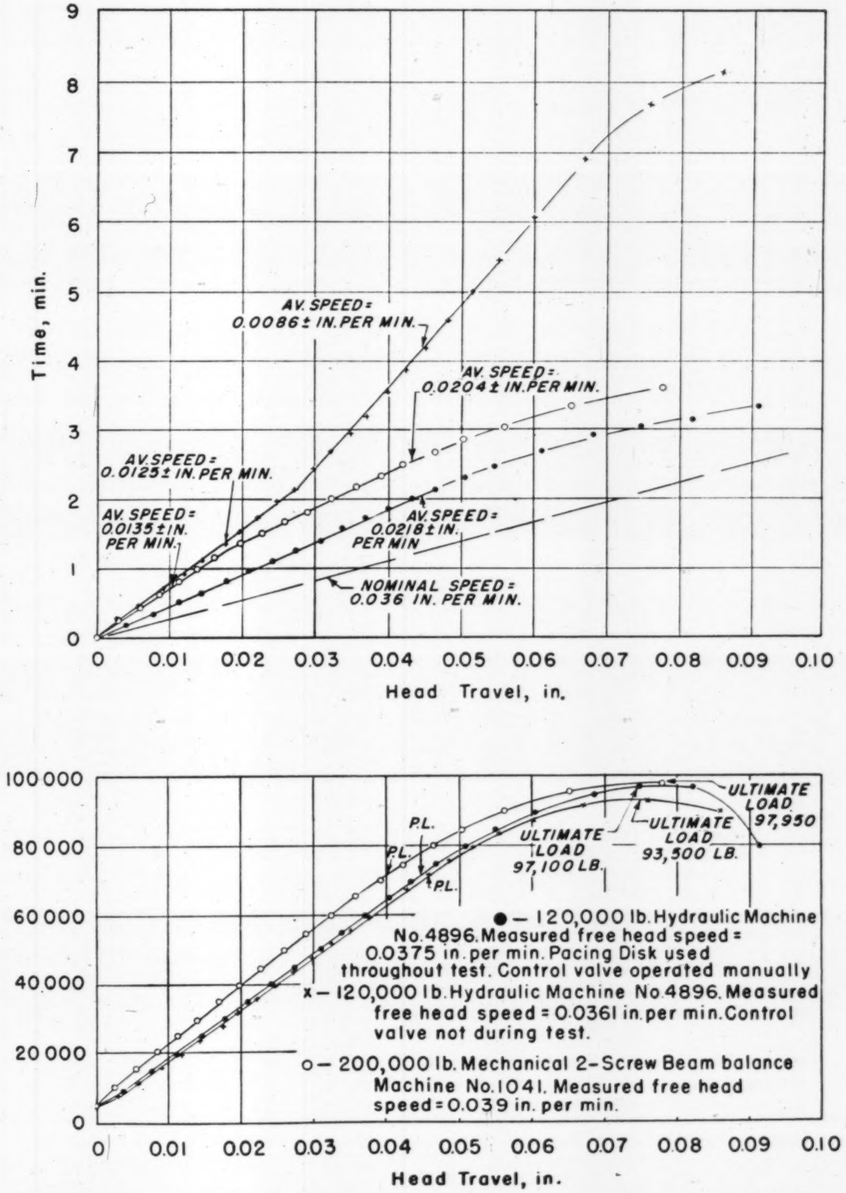


FIG. 4.—Comparison of the Rate of Loading Characteristics of a Mechanical- and a Hydraulic-Type Testing Machine for Tests of 3 by 5 by 12-in. Douglas-Fir Compression Specimens.

machine, the loaded crosshead speed is markedly reduced until the first ultimate load is reached, when the speed approaches the free-running crosshead speed of the testing machine. When the next shorter specimen begins to take load, the loaded crosshead speed decreases to a value approximately equal to the speed at the start of the test, increases as the load increases beyond the proportional limit, and approaches free running crosshead speed. This speed was not reached because specimen No. 3 began to take load before maximum load was reached on specimen No. 2. After all three specimens had been tested to maximum load, the loaded crosshead speed was approximately equal to the free-running crosshead speed. The curves also show the small change in deformation with time that occurred when the 1800-lb. loading head was transferred from one screw surface to another on the loading screws and the subsequent nonelastic deformation took place.

In contrast to the speed-time curve for the mechanical machine is the one obtained for the similar test in the hydraulic-type testing machine with the use of a rate-of-strain pacer. This latter curve is essentially a straight line throughout the test with an average value approximately equal to the free-running crosshead speed.

In a third series of tests by Youngquist and Liska, a comparison was made of the operating characteristics of a 120,000-lb. capacity testing machine of the packed-ram hydraulic-loading type and several mechanical-type testing machines of appropriate capacities, depending on the strength of the specimen under test. The hydraulic testing machine was operated using a rate-of-strain pacer and manual operation of the loading control valve to control the rate of loaded crosshead movement and also at a constant setting of the control valve at a value determined from free-running head speed tests. The data from this test series are shown in Figs. 2, 3, and 4, wherein the load-head travel curves are shown in the lower portion of the figures with the related time-head travel curves plotted above.

The procedure for test was as follows: In the case of the mechanical machines a gear ratio was selected to give a free-running head speed in agreement with that prescribed in Tentative Methods of Testing Small Clear Specimens of Timber (D 143 – 47 T)[3] Several no load test runs were made while measurements of head travel and time were obtained to secure a measured value of free-running head speed. The test specimen was then placed in the testing machine and simultaneous readings of head travel, load, and time were taken as the specimen was progressively loaded to failure. A typical plot of the data obtained is given in Fig. 2.

Before the tests of matched specimens were made in the hydraulic testing machine, several runs under load were made to insure uniform operating conditions. Then several free-running head speed tests were made, and measurements of head travel and time were taken as the loading control valve was adjusted until the desired free-running head speed was attained. Without disturbing the loading valve, the release valve was opened and the test specimen placed in the machine. The release valve was then closed and the specimen tested to failure at a rate of movement of the loaded crosshead directly related to the free-running head speed. No reference was made to the rate-of-strain pacer, and simultaneous readings of load, head travel, and time were taken to maximum load on the specimen. The third matched specimen in each set was tested in the hydraulic machine that was operated by manually adjusting the loading valve in accordance with the action of the rate-of-strain pacer.

---

[3] Tentative Methods of Testing Small Clear Specimens of Timber (D 143 – 47 T), 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 324.

Figure 2 shows the data obtained from tests of three matched 1 by 1 by 4-in. Douglas-fir compression-parallel-to-grain specimens tested according to the procedures outlined previously. The strength characteristics for all three specimens are similar despite a considerable variation in rate of loading during the initial part of each test. The average loaded crosshead speed, for the test of the specimen tested in the hydraulic testing machine operated in accordance with the demands of the rate-of-strain pacer, is very close to the measured free-running crosshead speed, shown by the light straight line on the graph. The average speed for the specimen tested in the hydraulic machine operated with a fixed loading valve opening is about 70 per cent of the free-running head speed, while the specimen tested in a 10,000 lb. mechanical-type testing machine was loaded at a rate less that 50 per cent of the free-running head speed. After the proportional limit had been exceeded, however, the loaded crosshead speed in each test was approximately equal to the free-running head speed.

Similar data are presented in Fig. 3 for tests of three matched Douglas fir compression-parallel-to-grain specimens 3 by 4 by 12 in. in dimension, and the same tendencies are indicated as were evident in the tests of the smaller specimens. One change of importance, however, is that the loaded crosshead speed for the specimen tested in the hydraulic testing machine operated in conjunction with the rate-of-strain pacer was only about two-thirds of the measured free-running head speed for loads less than the proportional limit, since the load modulus for these tests was much higher. Above this load the head speeds tended to be equal. The time-head travel plots for the matched specimens tested in the hydraulic testing machine with constant control valve opening and in a 100,000-lb. capacity mechanical-type testing machine are similar for loads less than the proportional limit. Up to this point the loaded crosshead speed was about one-third of the measured free-running head speed. Beyond this point, however, the loaded crosshead speed for the mechanical testing machine tended to increase and approached the free-running crosshead speed, while the head-speed in the hydraulic testing machine decreased rapidly. This would indicate that as the load approaches the capacity of the hydraulic-type testing machine, the movement of the piston or hydraulic ram decreases unless the gate valve is opened.

These observations are emphasized by similar data obtained in tests of three matched Douglas fir compression specimens 3 by 5 by 12 in. in size that are plotted in Fig. 4.

One other study was made to emphasize further the need for and use of rate-of-strain pacers on hydraulic-type testing machines. Two matched Douglas fir compression-parallel-to-grain specimens 3 by 4.5 by 12 in. in dimension were tested in a 100,000-lb. capacity hydraulic testing machine of the lapped-ram type after preloading to insure uniform operating conditions and to permit observations to determine the free-running head speed. One specimen was tested to failure as the machine was operated in conformance with the performance of the rate-of-strain pacer, and the data (Fig. 5) show that the loaded crosshead speed was about 50 per cent of the free-running head speed for the initial part of the test. Again

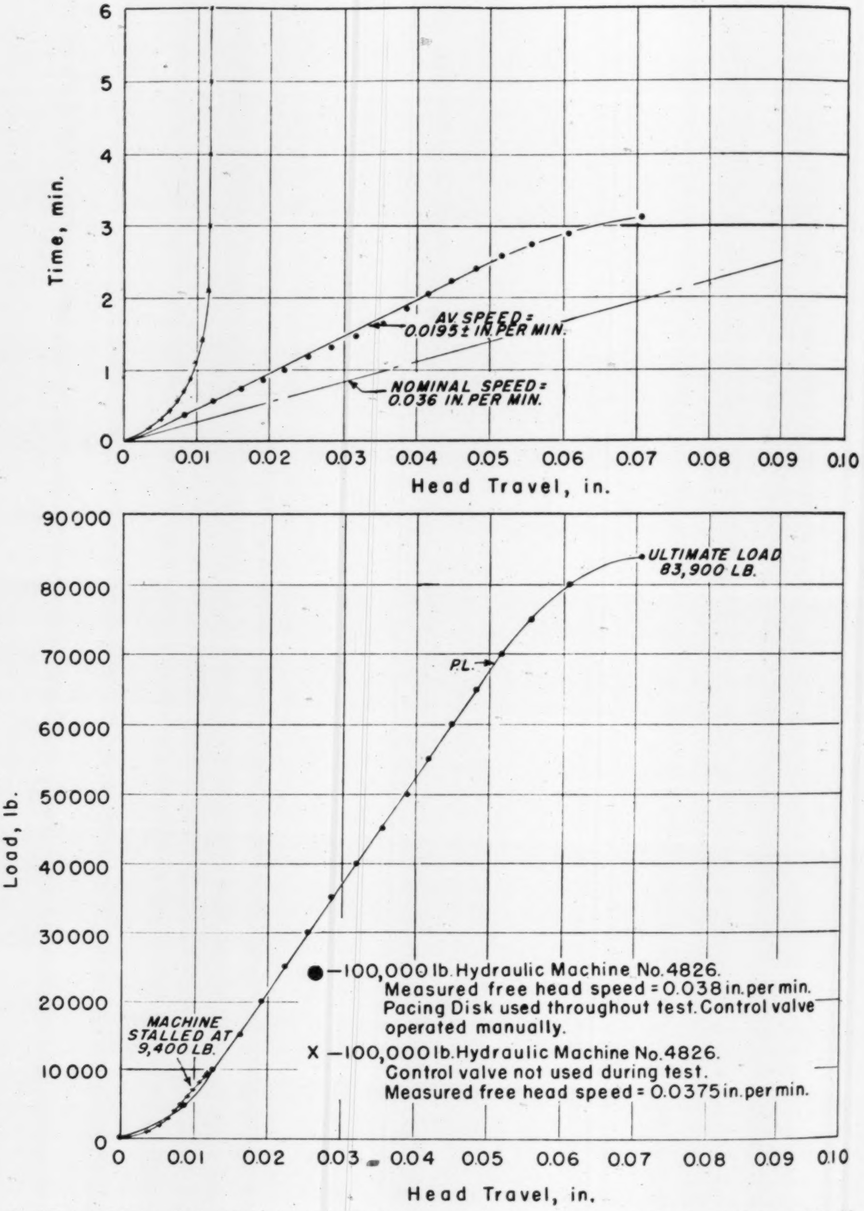


FIG. 5.—Comparison of the Rate of Loading Characteristics of a Hydraulic-Type Testing Machine for Tests of 3 by 4.5 by 12-in. Douglas-Fir Compression Specimens Under Two Different Operating Conditions.

the crosshead speed increased as the load exceeded the proportional limit value. When a test of the second specimen was made with the testing machine operated at a fixed control valve setting, equal to that used in obtaining the desired free-running head speed, the machine stalled at 9400 lb.

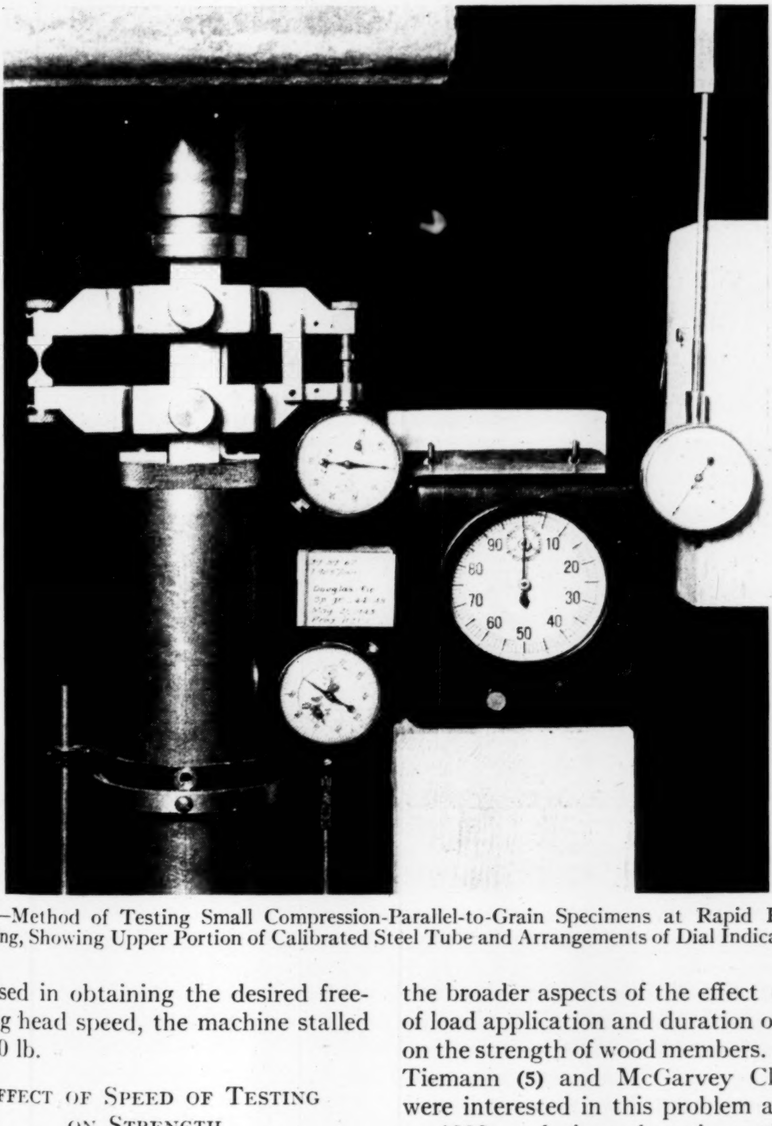


FIG. 6.—Method of Testing Small Compression-Parallel-to-Grain Specimens at Rapid Rates of Loading, Showing Upper Portion of Calibrated Steel Tube and Arrangements of Dial Indicators.

EFFECT OF SPEED OF TESTING ON STRENGTH

While some studies at the Forest Products Laboratory have been directed toward establishing the causes of variation in actual testing speeds in the interest of greater standardization of test conditions among investigators, considerable research has been conducted on the broader aspects of the effect of rate of load application and duration of stress on the strength of wood members. H. D. Tiemann (5) and McGarvey Cline (2) were interested in this problem as early as 1908, and since that time other investigators have expanded and elaborated on these studies. Recent studies have been concerned both with the effect of rapid loading, with elapsed time to failure (in fractions of a second), and the effect of duration of stress to determine the magnitude of the load that may be carried for different periods of time. The evaluation of the effect of rapid loading with respect to compression and bending is particularly applicable to aircraft design, and the duration studies are of primary importance in establishing working stresses for use in structural design. All of these studies are related and will provide data that will establish the strength of wood under extremely short as well as long-time loading conditions. This entire study, however, is beyond the scope of this paper, wherein the discussion will be limited to a single phase of the broader problem and will be concerned only with strength variations due to variations in speed of static testing machines. The effect of this variation in rate of loading on the compression-parallel-to-grain and flexural strengths of small clear specimens of Sitka spruce and Douglas fir was investigated by M. P. Brokaw and G. W. Foster (1) in 1945.

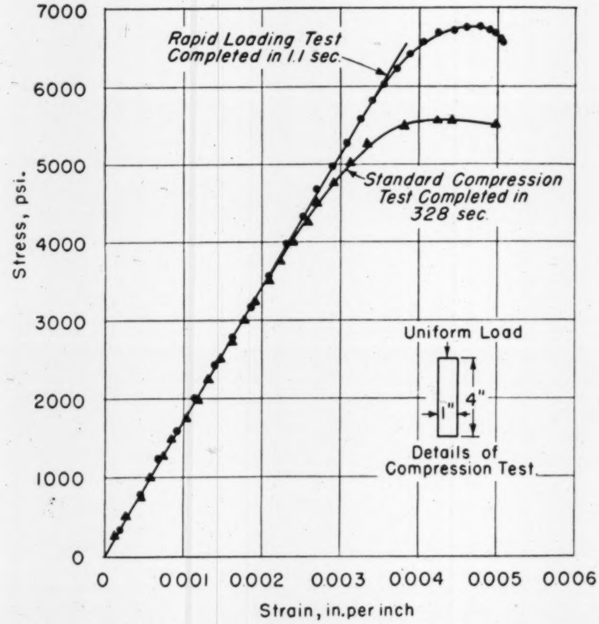


FIG. 7.—Stress-Strain Curves for Compression-Parallel-to-Grain Tests of Two Matched Douglas-Fir Specimens at Rapid and Normal Rates of Loading.

The compression-parallel-to-grain test specimens were nominally 1 by 1 in. in cross-section and 4-in. long. They were conditioned to constant weight prior to test at 74 F. and 65 per cent relative humidity. The matching plan was so arranged that one specimen for evaluation of rate-of-loading effect was oriented longitudinally parallel to the grain direction between two control specimens. The control specimens were tested in accordance with the requirements of Methods D 143 – 47 T,[3] as modified for the small-sized specimen. The strength of the rapid loading specimen was assumed to be equal to the average strength of the two end-matched controls in determining the effect of the faster rate of loading. Specimens were loaded at nominal rates of free-running crosshead movements of 0.087, 0.39, 0.77, 1.90, and 2.80 in. per min.

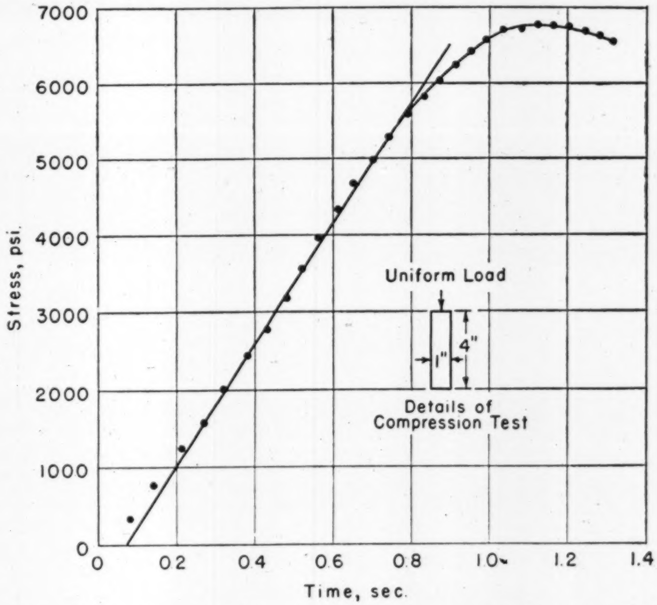


FIG. 8.—Stress-Time Curve for a Rapid Loading Test of a Douglas-Fir Compression-Parallel-to-Grain Specimen.

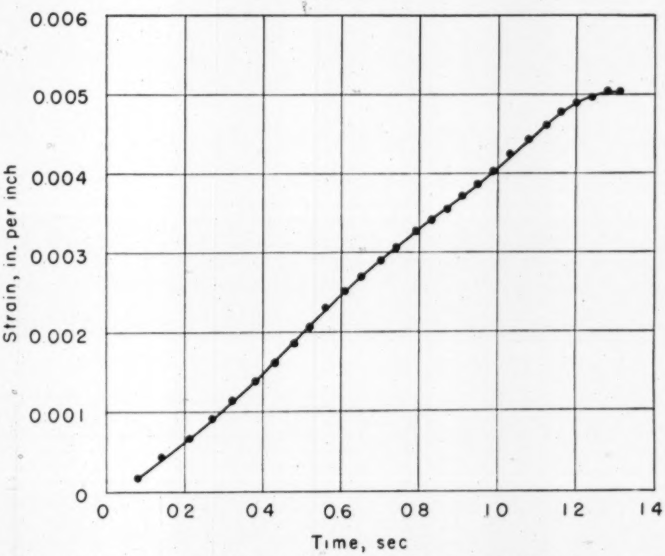


FIG. 9.—Strain-Time Curve for a Rapid Loading Test of a Douglas-Fir Compression-Parallel-to-Grain Specimen.

At these rapid rates of loading it was not possible to measure values of load and deformation in the manner used for static tests. The load was measured instead by means of the deformation of a

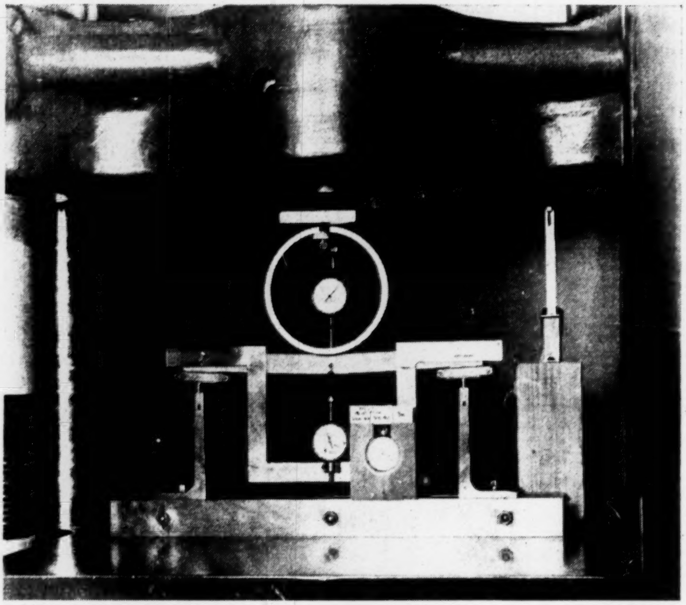


FIG. 10.—Method of Testing Small Sitka Spruce Flexure Specimens Loaded in 1.5 to 126 sec. The specimen with deflectometer attached is shown below the calibrated steel ring which serves as a loading device.

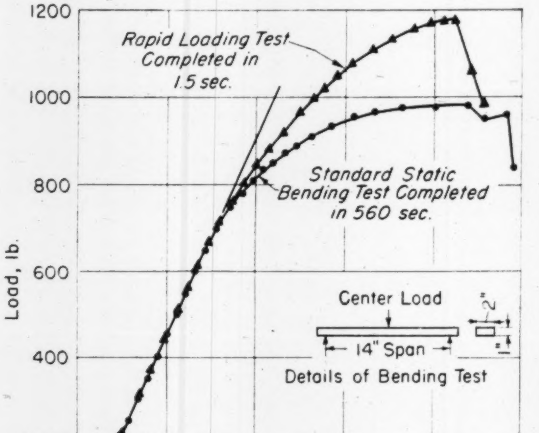


FIG. 11.—Load-Deflection Curves from Flexure Tests of Matched Sitka Spruce Specimens Tested at Rapid and Normal Rates of Loading.

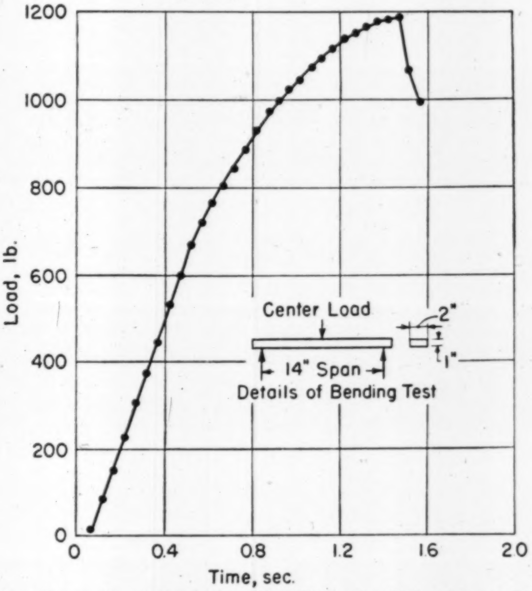


FIG. 12.—Load-Time Curve for a Rapid Loading Test of a Sitka Spruce Flexure Specimen.

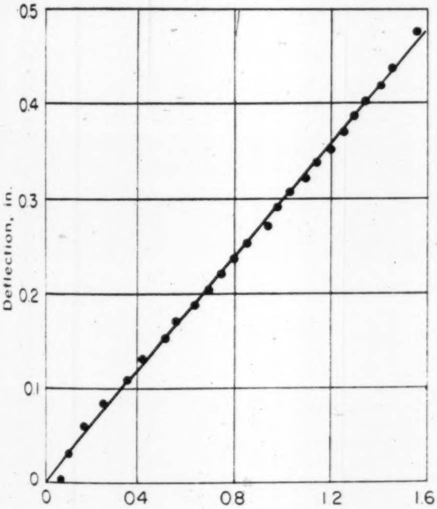


FIG. 13.—Deflection-Time Curve for a Rapid Loading Test of a Sitka Spruce Flexure Specimen.

calibrated steel tube that supported the specimen in the testing machine as shown in Fig. 6. Strain measurements were made by means of an especially developed compressometer having a dial gage reading to 0.001 in., and intervals of time were measured with an electric timer graduated to 0.01 sec. Since the rapid loading rates prohibited normal visual reading of these dials for load, deformation, and time increments during test, a special technique employing a high-speed, 35-mm. motion picture camera was developed. This camera was operated at an appropriate constant rate of film speed to record the movement of the dials and gages. The data were later transcribed from the film.

The percentage increase in strength due to rapid application of load was obtained by comparing the strength of the test specimen with that of the average of the two end-matched controls. Such a comparison for a typical specimen of Douglas fir is shown in Fig. 7. The stress-strain curve for the specimen tested in 1.1 sec. is compared to that of one of the matched controls. Characteristic curves showing the relationship between stress and time, and strain and time, for this same specimen are shown in Figs. 8 and 9.

In the study of the effect of rate of loading on properties in transverse bending, Douglas fir and Sitka spruce specimens 1 by 2 in. in cross-section and 16-in. long were used. The annual rings were parallel to the 2-in. face. The matching and conditioning procedures for the flexure specimens were similar to those for the compression-parallel-to-grain specimens, and, after conditioning to constant weight, the control specimens were tested in static bending under a centrally applied load on a 14-in. span. The test procedure was similar to that

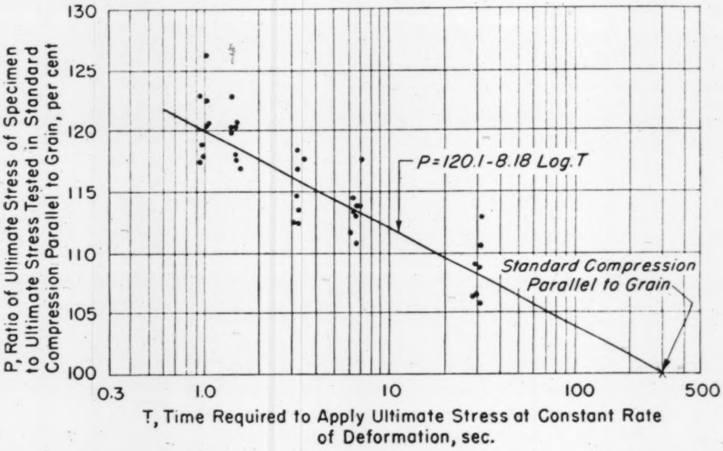


FIG. 14.—Effect of Rapid Loading on the Ultimate Strength of Douglas-Fir Compression-Parallel-to-Grain Specimens 1 by 1 by 4 in. in Dimension.
Each point represents an individual test.

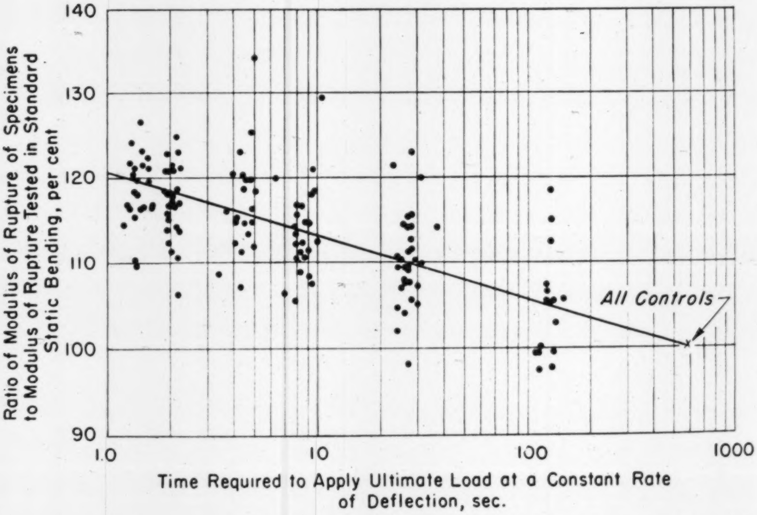


FIG. 15.—Effect of Rapid Loading on the Modulus of Rupture of Sitka Spruce Flexure Specimens. Specimens 1 by 2 by 16 in. in Size Tested Flatwise on a 14-in. Span.
Each point represents an individual test.

outlined in A.S.T.M. Methods D 143–47 T,[3] modified for the small-sized specimen. The rapid loading tests were made at nominal rates of free-running crosshead movement of 0.2, 1, 3, 6, 12, and 18 in. per min., and the properties obtained were compared with the average properties of the end-matched controls.

It was necessary to modify the standard test procedure for the rapid loading tests. To avoid the inertia effects of the testing machine weighing system, the load was determined by measuring the deflection of a specially designed, calibrated steel ring, similar to a proving ring, that was substituted for the center loading block. Deflections at the center of the beam were measured with a 0.001-in. dial gage held by a yoke that was attached to the specimen at the neutral axis above the supports. Time intervals to 0.01 sec. were measured by means of a stop watch. As with the compression-parallel-to-grain test, a high-speed motion picture camera was used to record load, deflection, and time intervals during the test. A general view of the test setup is given in Fig. 10.

Typical data from a test of a Sitka spruce flexure specimen tested in 1.5 sec. are plotted in Fig. 11 together with data from one of the matched controls. The relationships between load and time and deflection and time for these same test specimens are plotted in Figs. 12 and 13.

An examination of the stress-strain and load-deflection curves for the compression and flexure specimens, Figs. 7 and 11, tested at rapid rates of loading will serve to illustrate the relationships between properties obtained at rapid and normal rates of loading. In these specific instances, the ultimate strength is increased 22 and 20 per cent, respectively, when the load is applied in 1.1 and 1.5 sec. The proportional limit values are similarly increased, but the moduli of elasticity are little affected by rate of loading. The load-time curves are similar to the load-deflection curve, and the strain-time curves tend toward straight lines indicating an approximately uniform rate of deformation.

Data from the compression and flexure tests are plotted in Figs. 14 and 15 on a semilogarithmic scale and give the average relationships between ultimate strengths and rate of loading. The data are plotted as a ratio of test specimen strength to the average of that of the matched controls with respect to time required to ultimate load. Equations established on the basis of these data indicate that the relationship between rate of loading or speed of testing and strength are, for the species discussed:

Compression (Douglas fir)

$$P = 120.1 - 8.18 \log T$$

Flexure (Sitka spruce)

$$P = 120.7 - 7.56 \log T$$

where P = ratio of ultimate strength or modulus of rupture of the specimen tested to the average of the same property obtained on the matched controls tested at normal rates of head speed, and T = loading time in seconds.

Similar equations have been established for the other species tested under this program, and additional data on three hardwood species are being analyzed to determine these same characteristics. When combined with information being obtained on the effect of duration of long-continued loading, with loading periods up to 4 or 5 yr., the data will permit an accurate evaluation of the effect of rate and duration of loading on strength of wood over a wide range. Present data show an average decrease in strength of 6 to 10 per cent as the time of loading is increased by a factor of 10. This information is applicable to the use of wood and wood products in structural design, and also serves as a guide in the establishment of speed-of-test tolerances.

METHOD OF SPECIFYING SPEED OF TEST

In considering the problem of specifying speed-of-test requirements in the light of the background information presented on performance of testing machines, the following specification requirement[3] is currently used in connection with methods of testing wood and wood-base material:

> "The load shall be applied continuously throughout the test at a rate of motion of the movable crosshead of . . . . inches per minute." "The testing machine speed used shall not vary by more than plus or minus 25 per cent from that specified for a given test. If the specified speed cannot be obtained, the speed used shall be recorded on the data sheet. The crosshead speed shall mean the free-running or no-load speed of crosshead for testing machines of the mechanical drive type and loaded crosshead speed for testing machines of the hydraulic loading type."

It is difficult to prepare a specification which will apply equally and uniformly for all types of test materials and test equipment, and continuous study should be given to methods of further improvement. In connection with international developments in the standardization of methods of testing wood, it has been suggested as a means of limiting speed of test that the load shall be applied continuously and uniformly throughout the test at such a rate that the maximum load will be reached in not less than . . . . minutes.

This suggestion resolves the question of speed control into perhaps its simplest form while at the same time accomplishing the important achievement of unifying test results in so far as they are affected by speed of test. It also has the advantage that the speed of test may be retarded somewhat if necessary to obtain load-deformation data. This may be done without greatly lowering the test values, inasmuch as it has been shown by the data presented herewith that doubling the speed of test on wood causes about a 3 per cent increase in maximum load. The suggested control method may not be applicable to all materials, but the proposal is worthy of serious consideration because of its simplicity, ease of application, and flexability.

CONCLUSIONS

Wood is included among the materials in which the mechanical properties are materially influenced by rate of loading and duration of stress and for which specifications limiting the speed of testing are essential.

Compression-parallel-to-grain and flexure tests made under normal and rapid loading conditions, combined with long-time tests, indicate that the ultimate strength increases 6 to 10 per cent with tenfold increase and about 3 per cent with twofold increase in rate of loading.

The loaded crosshead speed in both the mechanical and the hydraulic-drive type of testing machines is influenced by elastic and nonelastic deformations of the testing machine itself and by nonelastic deformations of specimen fittings.

The loaded crosshead speed is largely a function of the load-modulus (or total stiffness of the test specimen) and the elastic deformations of the testing machine, it being assumed that nonelastic deformations (except slip in grips) are removed early in the test. The elastic deformation will vary among testing machines and in the same testing machine, depending on the effective length of the loading screws. Aside from elastic deformation, the load itself affects the loaded crosshead speed only when it approaches a balance with the ram or piston pressure available with a fixed control valve opening particularly in a lapped-ram hydraulic-loading type testing machine.

A rate-of-strain pacer operated in conjunction with a hydraulic-type testing machine, and also with a mechanical-type machine having a continuously variable speed control, is the most effective means of keeping the loaded crosshead speed approximately equal to the free-running crosshead speed. The rate-of-strain pacer would appear to be more effective if operated between the heads of the testing machine instead of between the loading head and the base of the machine as is now common practice.

In all studies made, except that of the hydraulic testing machine of the lapped-ram type operated at a fixed gate valve opening in the upper ranges of machine capacity, the loaded crosshead speed tended to approach free-running crosshead speed at stresses above the proportional limit of the wood. Tests have shown that the modulus of elasticity is not affected by rate of loading. If it can then be assumed that the influence of rate of loading is greatest in the range between the proportional limit and ultimate, it follows from this characteristic of testing machines that the actual effect on test results involved in the use of the free-head speed as a means of speed control is less than seems apparent.

It is apparent from these observations that specification requirements for free-running crosshead speed cannot be regarded as equivalent to the loaded crosshead speed and that even the loaded crosshead speed as controlled by a rate-of-strain pacer may vary greatly from the desired value. While in each case the actual rate of crosshead motion is increased as the free-running head speed is increased, the net effective speed is dependent on many factors that cannot be taken into account or compensated for in the calibration of a testing machine.

The presently used method of specifying rate of loading for wood and wood-base materials, taking into consideration the testing machine characteristics, is as follows:

> "The load shall be applied continuously throughout the test at a rate of motion of the movable crosshead of . . . . in. per min." "The testing machine speed used shall not vary by more than plus or minus 25 per cent from that specified for a given test. If a specified speed cannot be obtained, the speed used shall be recorded on the data sheet. The crosshead speed shall mean the free-running or no-load speed of crosshead for testing machines of the mechanical drive type and loaded crosshead speed for testing machines of the hydraulic loading type."

Attention is called to a possible simpler method of expressing rate of loading limitations, which has been proposed for consideration as follows:

> "The load shall be applied continuously and uniformly throughout the test at such a rate that the maximum load will be reached in not less than . . . . minutes."

REFERENCES

(1) M. P. Brokaw and G. W. Foster, "Effect of Rapid Loading and Duration of Stress on the Strength Properties of Wood Tested in Compression and Flexure," *Forest Products Laboratory Report No. 1518* (1945).

(2) McGarvey Cline, "Forest Service Tests to Determine the Influence of Different Methods and Rates of Loading on the Strength and Stiffness of Timber," *Proceedings*, Am. Soc. Testing Mats., Vol. VIII, p. 535 (1908).

(3) W. Kendrick Hatt, "Instructions to Engineers of Timber Tests," *Forest Service Circular 38*, U. S. Dept. Agriculture (1906).

(4) L. J. Markwardt, Round Table Discussion on Speed of Testing, held at the Fifteenth Annual Meeting of the A.S.T.M., Atlantic City, N. J., June 18, 1947 (1947). (Available in mimeographed form.)

(5) Harry D. Tiemann, "The Effect of the Speed of Testing upon the Strength of Wood and the Standardization of Tests for Speed." *Proceedings*, Am. Soc. Testing Mats., Vol. VIII, p. 541 (1908).

EFFECT OF SPEED OF TEST UPON STRENGTH PROPERTIES OF PLASTICS*

By A. G. H. Dietz,[1] W. J. Gailus,[2] and S. Yurenka[2]

Strength properties of plastics are, in general, sensitive to both time and temperature. In some plastics the time-dependence is more marked than in others; time-dependence is affected by temperature and considerable influence is exerted by fillers and laminates, but the basic influence of time and, as a consequence, the rate at which a test is made, is a factor of fundamental importance.

Numerous investigators have reported upon the effect of the speed of test upon the strength properties of various plastics. No exhaustive review can be attempted here but a few examples will be cited to show the general trends. Emphasis will be placed upon tests carried out in the "static" range, but brief mention will also be made of fatigue, creep, and tests at high speeds. This review will be followed by a brief exposition of some of the theories of visco-elastic or plasto-elastic behavior, a short discussion of the basic concept of the true stress - true strain relationship as opposed to the ordinary stress - ordinary strain relationship usually employed, and finally a description of tests carried out at constant rates of ordinary and true strain on a variety of plastics materials ranging from soft and extensible to hard and relatively unyielding. Some of the testing equipment and extensometers especially developed for these tests will be described.

W. F. Bartoe (**5, 6, 7**)[3] and D. S. Frederick(**7**) showed that the stress-strain behavior of polymethyl methacrylate (Plexiglas I) varied with the rate of straining (Fig. 1). Tensile strength varied from 5500 to 7000 psi. as the rate of straining was increased from 0.050 to 0.400 in. per min. at 40 C. Similarly, when "dead-load stressed" at initial stresses varying from 3500 to 7000 psi., the time to reach 10 per cent deformation in compression on 0.5-in. cubes at 40 C. varied from 50 min. to approximately 660 hr. R. N. Haward (**36, 37**) made tests of polymethyl methacrylate incorporating 25 per cent dibutyl phthalate. These were made at constant loads ranging from 60 to 130 kg. per sq. cm., and the family of curves shown in Fig. 2 was obtained. This is a typical set of short-time creep curves. W. N. Findley (**22, 23, 24, 25**) has reported the effect of varying rates of extension upon the stress-strain relations in tension, compression, and torsion of cellulose acetate plasticized with about 26 per cent aromatic phosphate esters. Rates of strain were correlated with rates of cross-head motion by timing the strain intervals on the extensometers. Some of these results are summarized in Figs. 3, 4, and 5. Findley concluded in part:

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Associate Professor, Department of Building Engineering and Construction, Director of Plastics Research Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

[2] Research Associate and Research Assistant, respectively, Plastics Research Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

[3] The boldface numbers in parentheses refer to the Selected Bibliography appended to this paper, see p. 1183.

"Yield point and fracture stress were found to increase with increasing rate of strain for tests in tension, compression, and torsion.

"Modulus of elasticity in tension and torsion was found to be independent of rate of strain for values tested, but modulus in compression was found to increase with increasing rate of strain for loading in compression. The following values of modulus were found: Tension, $E = 244{,}000$ psi.; torsion, $G = 78{,}000$ psi.; compression, E varied from 204,000 to 278,000 psi.

"At a tensile rate of strain of 0.04 per min., the values of upper yield point were: Tension, 4400 psi; torsion, 3300 psi.; compression (long specimen), 4300 psi. And the fracture stress was: Tension, 5500 psi.; and torsion (modulus of rupture), 4900 psi.

"Aging of material at constant temperature and relative humidity was found to increase tensile strength about 4 to 12 per cent and modulus of elasticity in tension about 15 per cent during about one year."

In repeated-bending fatigue tests on the same cellulose acetate with a constant amplitude of deflection cantilever machine, operating at frequencies ranging from 42 to 2900 cycles per minute, Findley found that the endurance limit, adjusted to an age of 11,000 hr., dropped from a computed value of 2000 psi. at 42 cycles per minute to 1400 psi. at approximately 750 cycles per min. and thereafter remained substantially constant over the range 750 to 2900 cycles per min. (Fig. 6). In these tests it was necessary to keep the specimens cooled with an air blast. Otherwise, excessive rises in temperature caused softening of the material. It was postulated that relaxation of stress might have occurred during the individual cycles at lower frequencies. Findley also found that the fatigue behavior of a laminated Mitscherlich-paper plastic depended upon the frequency. These tests were conducted on rotating cantilever-beam specimens at constant amplitude of alternating stress and at frequencies ranging from 500 to 14,000 rpm. In general, the fatigue strength at a given number of cycles decreased with increasing frequency. At the higher frequencies, the measured temperatures of the specimens increased, in some instances to above 150 F. (14,000 rpm. and fatigue failure at 10^4 cycles). The effect of temperature, consequently, was operative as well as the effect of rate of test.

C. H. Adams in an unpublished communication gives values of tensile strengths as substantially linear functions of logarithm of rate of straining (Fig. 7) for polystyrene (Lustron), heat-resistant polystyrene (Lustrex), and heat-resistant styrene copolymer (Cerex 250).

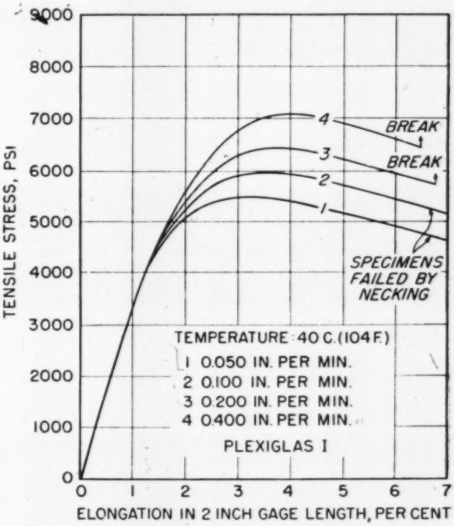


FIG. 1.—Effect of Rate of Strain on Strength of Plexiglas I (Bartoe and Frederick).

T. S. Carswell and H. K. Nason (**16, 17**) have also reported a linear relationship between the logarithm of the rate of straining and the tensile strength of injection molded polystyrene and cellulose acetate (Fig. 7). These same authors, with T. S. Lawton (**51**) found that cellulose nitrate gave a curvilinear relationship, rising with increased rate of strain. Adams determined the straining rates by timing the elongation, the other rates were based upon the free-running speed of the cross-head of the machine.

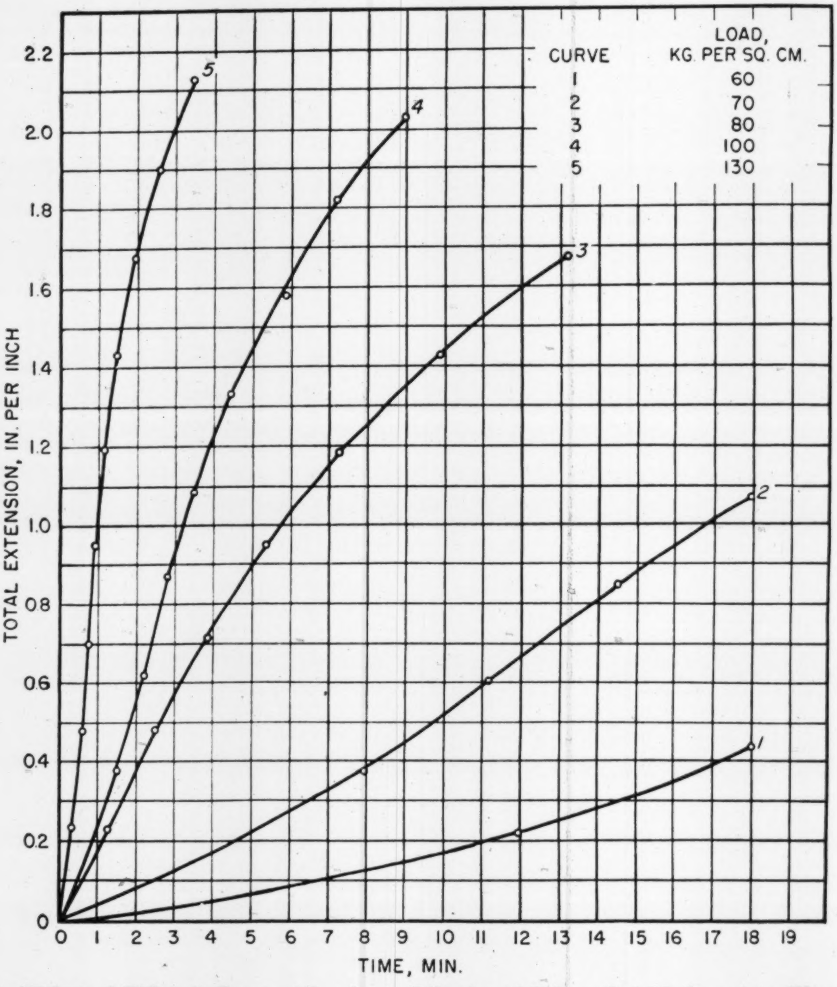


FIG. 2.—Effect of Different Constant Loads on Behavior of Polymethyl Methacrylate (Haward).

J. Miklowitz (68) found that in testing nylon the yield-point elongation increased three times for a 10,000-fold increase in the speed of stretching, and that the true yield stresses and yield load stresses increased almost two times over the same range. In nylon, the first and any succeeding sharp drops in load were accompanied by a constriction or "neck" forming a double yield front. As the speed of deformation increased the length of the advancing yield front decreased, that is, the maximum strain of a cross-section occurred more abruptly.

D. Telfair and H. K. Nason (79, 80) have discussed and made measurements of the energy required to fracture plastics specimens during impact tests. They point out that in plastics, as in other materials, ductile or brittle failures may be expected depending upon tem-

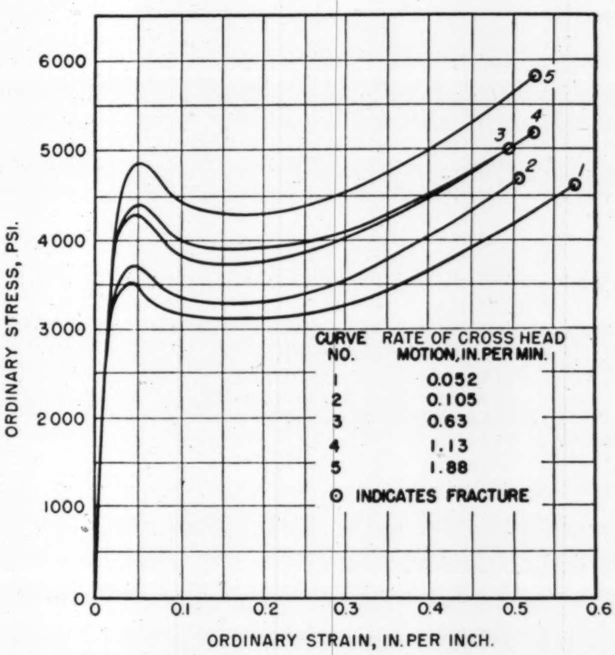


FIG. 3.—Rate of Strain *versus* Tensile Strength of Cellulose Acetate (Findley).

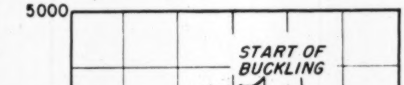


FIG. 4.—Rate of Strain *versus* Compressive Strength of Cellulose Acetate (Findley).

perature; the rate of test, and whether the critical tensile stress is reached before the critical shear stress required to cause yielding. D. R. Morey (69) also discusses the factors which influence the appearance of ductile or brittle fractures and shows that the speed of test and temperature are both critical.

C. S. Myers (71) has made a study of critical impact velocity *versus* sheet thickness of three copolymer vinyl chloride-acetate sheet stocks. L. H. Callendar (14) and others have studied the effect of variable height of drop upon the impact strength of plastics. These investigations all show that the stress values as well as the types of failures are influenced by the velocity of the test.

Creep properties of plastics have been studied by a number of investigators in addition to those already mentioned. J. Marin (60) made torsion and tension creep tests on cloth laminates at stresses ranging from 3010 to 6400 psi. in tension and 1200 to 5000 psi. in torsional shear. W. N. Findley (23, 24) carried out creep tests on cellulose acetate and macerated phenolic molding materials among others. D. Telfair, C. H. Adams, and H. W. Mohrman (78) made creep and long-time tensile and flexural fatigue tests of melamine and phenolic plastics. W. Gailus and D. Telfair (30) found that in thermosetting materials the shrinkage, produced by continuing "curing," might easily exceed the extension caused by creep. Others have reported similar

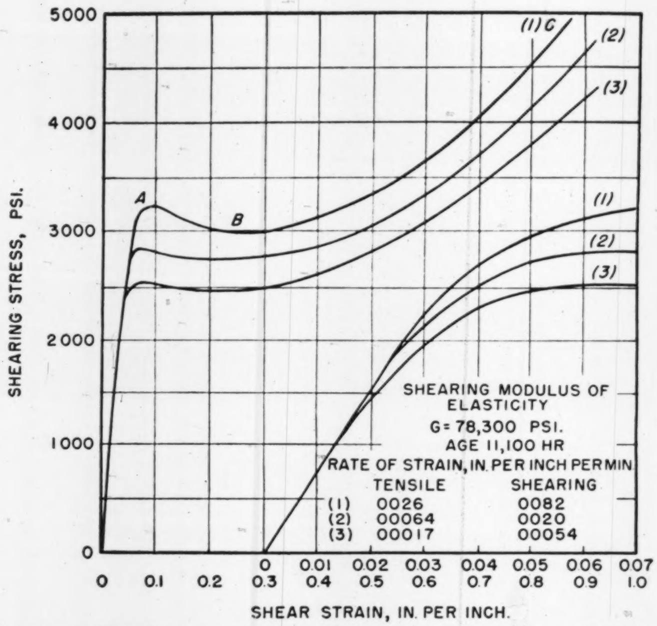


FIG 5.—Rate of Strain *versus* Shearing Strength of Cellulose Acetate (Findley)

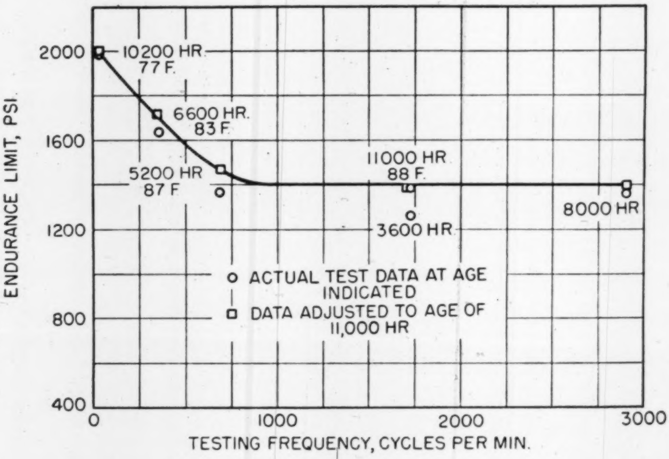


FIG. 6.—Speed of Test *versus* Endurance Limit of Cellulose Acetate (Findley).

phenomena. If these factors are separated out, however, the creep curves of plastics are similar to those of other materials, like metals, at least over the time intervals so far reported. Only a few of the plastics tests have approached the periods employed in some very long-time metals tests.

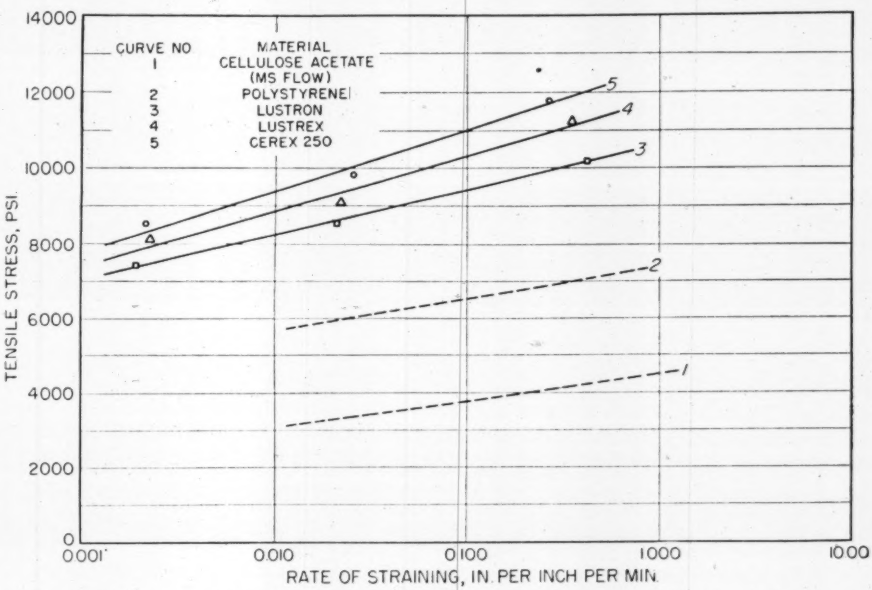


FIG. 7.—Rate of Strain *versus* Ultimate Tensile Strength of Several Plastics (Adams, Carswell, Nason).

Theory of Visco-elastic Behavior.—Mechanical Models:

It has long been recognized that many materials are not truly elastic and that time is an important factor in their behavior. H. Leaderman (**52, 53, 54, 55, 56**) has summarized much of the early work by W. Weber (**89**), F. Kohlrausch (**46, 47, 48**), R. Kohlrausch (**49**), and others who showed that upon load application many materials like glass, silk, and rubber, exhibited instantaneous deformation followed by a delayed "after effect" or continuing deformation. Upon release of load there was an instantaneous recovery followed by a continuing slow recovery which might or might not be virtually complete. Depending upon the speed of loading, either the instantaneous elastic or the delayed extensions might be predominant in the behavior of the material. The load-extension behavior, therefore, was dependent not only upon the magnitude of the load but also upon the rate at which it was applied.

Plastics behave similarly. Under stress they may exhibit the following:

1. Instantaneous extension (instantaneously recoverable),
2. Retarded extension (slowly but completely recoverable),
3. Flow (irrecoverable).

T. Alfrey (**2, 3**), H. Mark (**61, 62, 63, 64, 65**), E. Guth (**33, 34, 41, 42, 43**), H. M. James (**34, 41, 42, 43**), and others have shown that the relative importance of these three responses depends upon the molecular structure of the plastic, whether it is an amorphous linear high polymer or a more intricate cross-linked high polymer having a loose, tight, regular, or irregular network, containing material of low molecular weight or containing linear polymer molecules.

These authors and others have shown that linear high-polymers are more or less flexible because they tend to rotate about single valence bonds. As a result, many configurations are possible, of which a partially curled configuration is the most common. The diffusion of such a polymer is by chain segments (estimated by H. Eyring (**31**) to average 20 to 30 chain atoms in length) which "jump" to new equilibrium positions as "holes" open up in their vicinities, making new positions possible. This movement of chain segments is continually occurring and causes the polymer as a whole to wander about, in addition to constantly changing its shape. In the unstressed state, all motions are equally likely to occur in all directions and there is no change in shape of the whole. When a stress is applied, the motions are influenced or "biased" in the direction of stress. The first response to stress is an instantaneous, small, completely elastic separation. This is followed by a tendency to uncurl and pull out the kinked molecules in the direction of stress, that is, to change the partially curled normal configuration. At the same time the wandering of molecules as a whole is influenced to take place in the direction of stress. Both of these latter actions take time and the rate is not necessarily directly proportional to the stress. The change in configuration is reversed upon release of stress and eventually the molecules return to their normal partially curled configuration. This accounts for the retarded but completely reversible extension. The influenced motion of entire molecules past each other is not reversible and accounts for the true flow which is not recoverable.

In cross-linked three dimensional networks, the possibility of flow is greatly diminished or eliminated because the individual chains are not free to move past each other. The retarded extension may be small or great depending upon the distance between mesh points in the network, the degree of curl or kinkiness between mesh points, and the regularity or irregularity of the network.

Mechanical models are widely used to illustrate the behavior of high polymers. Perhaps the earliest and most widely used is that of J. C. Maxwell (**66, 67**) which consists of a spring and dashpot in series (Fig. 8 (*a*)). The spring, perfectly elastic, responds instantly to load; the dashpot, perfectly viscous in the Newtonian sense, has a retarded irrecoverable response whose rate is proportional to the stress applied. Together, their response is as shown, and the rate of deformation is given by the expression:

$$\frac{d\gamma}{dt} = \frac{1}{\eta} s + \frac{1}{G} \frac{ds}{dt} \quad \ldots\ldots(1)$$

where:

γ = displacement,
s = stress,
η = viscosity, and
G = shear modulus.

If the unit is extended quickly and held stationary, $\frac{d\gamma}{dt} = 0$.

and

$$\frac{1}{G} \frac{ds}{dt} + \frac{1}{\eta} s = 0 \quad \ldots\ldots\ldots(2)$$

which is solved:

$$s = s_o e^{-(G/\eta . t)} \quad \ldots\ldots\ldots(3)$$

After a time equal to $\frac{\eta}{G}$, the stress has decayed to $1/e$ of its original value. The ratio η/G is therefore called the "relaxa-

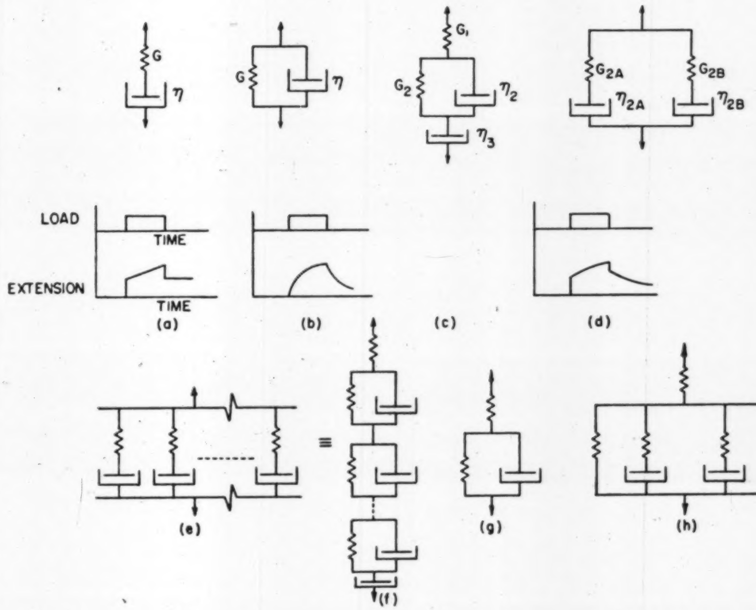


FIG. 8.—Mechanical Models of Behavior of Visco-elastic Materials.

tion time" and is usually designated as τ, therefore:

$$s = s_0 e^{-t/\tau} \ldots\ldots\ldots (4)$$

The Maxwellian element makes no provision for retarded recoverable extension. A model proposed by Voigt (Fig. 8 (*b*)) has a spring and dashpot in parallel whose response to load is as shown. The response to load is retarded by the dashpot, but when load is released the spring slowly pulls the element back to its original position. This model accounts for retarded elasticity but not for instantaneous elasticity and flow. The differential equation of its behavior is:

$$\eta \frac{d\gamma}{dt} + G\gamma = s \ldots\ldots\ldots (5)$$

If stress is applied over a period t, the deformation-time expression becomes:

$$\gamma = \frac{s}{G}\left(1 - e^{-(G/\eta t)}\right) \ldots\ldots (6)$$

The quantity η/G is called the "retardation time" τ, and Eq. 6 becomes:

$$\gamma = \frac{s}{G}\left(1 - e^{-(t/\tau)}\right) \ldots\ldots (7)$$

The simplest models expressing all three types of response (instantaneous extension, retarded recoverable extension, and flow) are those illustrated in Fig. 8 (c) and 8 (d). These can be shown to be equivalent. The behavior is as indicated.

G. J. Dienes (**20**), using a parallel plate plastometer, found that at elevated temperatures the behavior of vinyl chloride acetate, polyethylene, and cellulose ester resins could be depicted accurately by the simple mechanical model (Fig. 8 (c)) consisting of a retarded Voigt element in series with a pure elastic spring and a pure viscous dashpot. These materials are, therefore, completely characterized by two elastic moduli and two viscosity coefficients, each of which can be evaluated. High-temperature stress-strain curves at various rates of loading have been predicted theoretically and verified experimentally.

Plastics in general do not conform to the simple models of Figs. 8 (*c*) and 8 (*d*) because a large number of configurations and sizes of polymer chains may exist in a single sample. E. Wiechert (**88**) suggested that a model, such as shown in Fig. 8 (*e*), would express the actual behavior better. This, again, is equivalent to Fig. 8 (*f*).

It has been pointed out by Eyring, Halsey (**35**), Alfrey, and others that the springs involved in these various models are not necessarily Hookean (extension is not directly proportional to stress) and that the dashpots are not necessarily Newtonian (rate of flow is not directly proportional to stress). If all the elements in a polymer were linear (Hookean and Newtonian), sets of creep curves would be symmetrical in shape and would be equally displaced for equal increments of load. Haward's curves for polymethyl methacrylate (Fig. 2) show this not to be true, indicating that the elements are non-linear. Consequently, in load tests, it would not be expected that the response would be directly proportional to the rate of load.

In cross-linked three-dimensional network polymers in which flow is absent, mechanical models like that of Fig. 8 (*g*) have been proposed. Here the lower dashpot is omitted. Morey has also suggested a model like that in Fig. 8 (*h*). Here the left-hand spring is analogous to the skeletal structure brought about by the cross linking. The right-hand spring is a retractive force caused by the temperature motion of chain segments, such as is found in rubber.

True Stress-True Strain Relationships versus Ordinary Stress-Ordinary Strain:

The foregoing discussion of the visco-elastic or plasto-elastic behavior of high polymers, such as plastics, coupled with the above summarized results of tests by various investigators at various rates ranging from creep to high velocity raise some fundamental questions as to the best means of conducting tests on plastics, particularly those exhibiting considerable extensions. With the exception of the work reported by Miklowitz and some by Bartoe, the stresses and strains reported by the various investigators quoted above have been based upon the original cross-sections and original gage lengths of the materials, that is, the stresses and strains have been what are often referred to as "ordinary" stresses and "ordinary" strains.

P. Ludwick (**57**), as quoted by A. Nadai (**73**) showed that true tensile stresses, obtained by dividing the tensile load on a test specimen by the actual cross-section at the time the load acts, is in many ways a more meaningful value than the ordinary stress obtained by dividing the load by the original cross-section. If P is the load, A_o the original cross-sectional area, A the instantaneous cross-sectional area corresponding to a load P, L_o the original length, L the final length, and ϵ the unit extension defined as:

$$\epsilon = \frac{L - L_o}{L_o} \quad \ldots\ldots\ldots (8)$$

and if ordinary stress

$$s_o = P/A_o \quad \ldots\ldots\ldots\ldots (9)$$

the true stress may be obtained from the expressions:

$$S = \frac{P}{A}\ \frac{(1 + \epsilon)}{A_o} = (1 + \epsilon)S_o \ldots (10)$$

if the volume of the specimen can be supposed to remain constant ($A_oL_o = AL$).

The strain ϵ as defined above is the usual, or ordinary, strain commonly employed. Ludwik showed that the true strain is found by referring the extension at any instant to the actual length L at that instant instead of to the original length L_o, that is, over an extension L_o to L, the true strain is:

$$\epsilon_t = \int_{L_o}^{L} \frac{dL}{L} = ln \frac{L}{L_o} \quad \ldots(11)$$

C. W. MacGregor (**58, 59**) and his associates have studied the true stress-true strain relationship in considerable detail and have shown that it is much more significant than ordinary stress-ordinary strain when related to other forms of test such as combined stresses of various kinds, bending tests, fatigue tests, and impact tests. Furthermore, and this is of especial importance in plastics testing, the true stress-true strain relationship is necessary to a study of the laws governing the plastic flow and fracture of engineering materials. In the plastic range the strains are large and the reduction in cross-section correspondingly large, especially in materials that undergo necking.

When strains are small the difference between true and ordinary strain is small, but when they are large the difference is marked. For example, for extensions up to 25 per cent, the difference is less than 11 per cent, but for larger extensions the two values diverge rapidly. The effect upon typical stress-strain curves can be shown by mild steel whose ordinary stress-ordinary strain curve shows sharp changes at the yield points and rises to

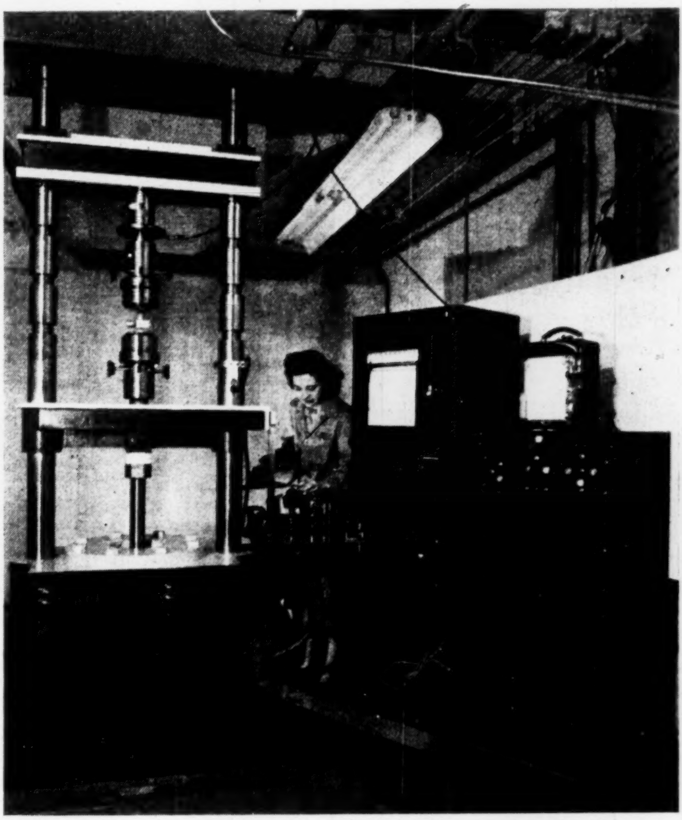


FIG. 9.—General View of Universal Plastics Testing Machine.

a maximum value after which it drops off. The true stress - true strain curve for mild steel has two principal parts, a straight line of one slope for the elastic portion and a second straight line of different slope for the plastic portion. These two are connected by a curve portion.

It may be expected that in plastics, where the effect of rate of strain has a marked influence upon the stress - strain relationships some differences in behavior will occur, depending upon whether a test is conducted at controlled rates of ordinary strain or controlled rates of true strain.

TESTS

Testing Equipment:

In order to study rate effects upon the strength properties of plastics, tests were carried out at constant rates of crosshead motion, constant rates of load application, and at constant rates of true strain and of ordinary strain. The testing machine employed has previously been described (12) and is illustrated in Fig. 9. Briefly, it incorporates servomechanisms which, operating through mechanical and electrical error-measuring circuits, control a variable-displacement pump which, in turn, moves a cross-head in such a manner as to provide the desired rates of crosshead motion, load, or strain.

Two extensometers were developed to make it possible to run tests at constant rates of either ordinary or true strain. These are shown in Figs. 10 and 11.

The "wheel" type of extensometer shown in Fig. 10 was developed as a control signal source for tests at constant rate of true strain or, conversely, to record true strain. It consists of a combination of knife-edge and a wheel attached to a potentiometer. The distance between the knife-edge and the wheel edge is fixed. As the material is extended it moves past the wheel causing the latter to rotate which develops a change in resistance in the potentiometer. The change in resistance is a measure of the distance the wheel has rotated and, consequently, is a measure of the amount of strain. To realize that this measured strain is really true strain (for materials which do not neck locally), one has merely to recall the definition of true strain as being the ratio of the instantaneous change in gage length to the instantaneous gage length, or, in the limit:

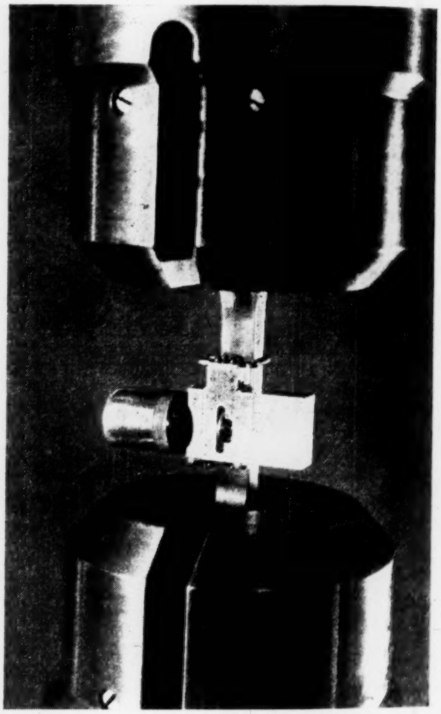


FIG. 10.—Detail, Showing Wheel-Type Extensometer.

$$\epsilon_t = \int \frac{dL}{L} \quad \ldots\ldots\ldots (12)$$

In the wheel extensometer L is constant and so may be removed from under the integral and so we are able to write:

$$\epsilon_t = \frac{1}{L}\int dL \ldots\ldots (13)$$

The change in resistance of the rotating potentiometer is linearly related to the term $\int dL$ so that we do get a measure of true strain. If the wheel rotates at a constant rate the material is being strained at a constant rate of true strain. The signal coming from the potentiometer is compared with a constantly increasing signal fed into the servomechanism which operates to keep the two signals alike and, therefore, to stretch the sample at a constant rate of true strain.

The "U-bar" extensometer shown in Fig. 11 consists of a pair of knife edges on the ends of the long arms of the U. Across the end of the U is a flexible strip of metal to which are attached two SR-4 type strain gages. As the material is stretched, the two ends of the bar are pulled apart, thereby bending the strip and developing strains in the strain gages. These strains are calibrated in terms of the extension of the sample. The U-bar extensometer yields ordinary strain rather than true strain. If, therefore, the signal is fed into the servomechanism, the sample is stretched at a constant rate of ordinary strain.

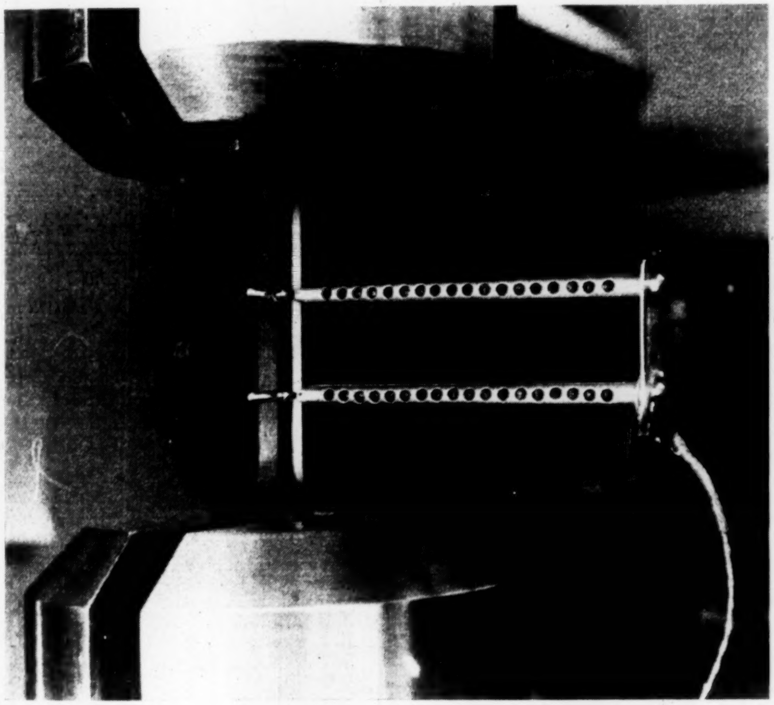


FIG. 11.—Detail, Showing U-Bar Extensometer.

Tests were run using both the wheel extensometer and the U-bar. References to tests at constant rates of true strain mean the wheel extensometer was employed; references to constant rates of ordinary strain mean that the U-bar extensometer was used.

Cellulose Acetate Butyrate:

Three grades of cellulose acetate butyrate, H4, MH, and S, hereafter referred to as hard, medium, and soft, were tested at constant rates of crosshead motion, constant rates of load, and constant rates of ordinary strain as shown in Figs. 12, 13, 14, and 15. The tests at constant rates of crosshead motion were plotted in terms of ordinary stress *versus* ordinary strain (Fig. 12) and true stress *versus* true strain (Fig. 13), the tests at constant rates of load were plotted in terms of ordinary stress *versus* ordinary strain (Fig. 14), and the tests at constant rates of ordinary strain were plotted in terms of true stress *versus* true strain (Fig. 15). The various rates employed for the tests were as indicated on the figures. As would be expected from the mathematics of the true strain and true

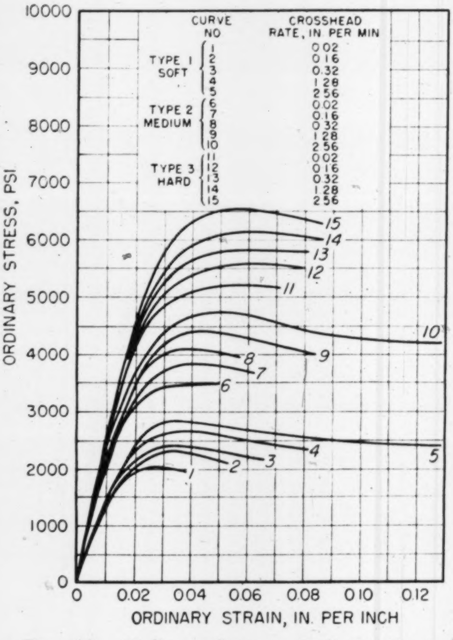


FIG 12.—Ordinary Stress - Ordinary Strain Curve in Tension; Cellulose Acetate Butyrate Types 1, 2 and 3; Tests at Various Constant Rates of Crosshead Motion.

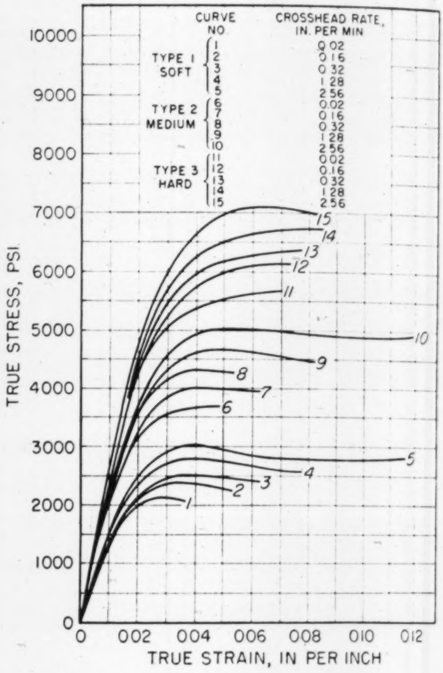


FIG. 13.—True Stress - True Strain Curves in Tension; Cellulose Acetate Butyrate Types 1, 2 and 3; Tests at Various Constant Rates of Crosshead Motion.

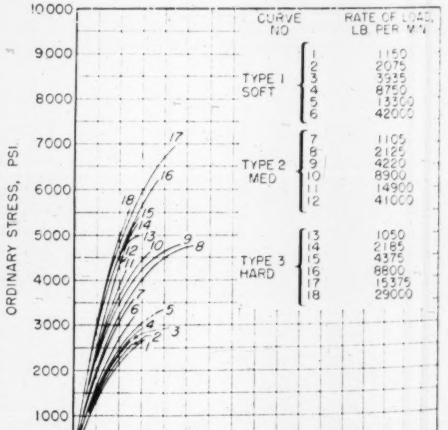


FIG. 14.—Ordinary Stress - Ordinary Strain Curves in Tension; Cellulose Acetate Butyrate Types 1, 2 and 3; Tests Run at Various Constant Rates of Load.

stress *versus* ordinary strain and ordinary stress (Eqs. 8, 9, 10, and 11), the curves of Fig. 13 are shifted upward and to the left of the curves of Fig. 12. True stresses are higher than ordinary stresses, and the corresponding true strains are lower. The apparent drop in the true stress - true strain curves is questionable since it is contrary to theory and to experience with materials other than plastics. The phenomenon may be attributable to slight unnoticeable necking, to experimental technique, or to the extreme difficulty of obtaining good results on rectangular specimens (made as specified in A.S.T.M. method D 638 – 46 T).[4] The same observations hold for all other curves showing an apparent drop in the true stress - true strain curves (Figs. 16, 17, 18, and 23).

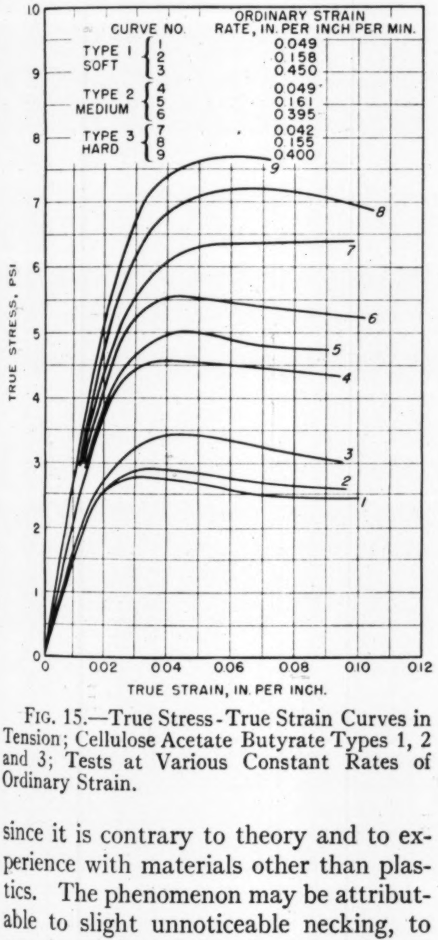


FIG. 15.—True Stress - True Strain Curves in Tension; Cellulose Acetate Butyrate Types 1, 2 and 3; Tests at Various Constant Rates of Ordinary Strain.

It may also be noted in these figures that the yield point and the moduli of elasticity (tangents at the bases of the curves) in general increase with increasing rate of crosshead motion. Crosshead rates were increased from 0.02 to 2.56 in. per min. for a speed ratio of 128 to 1. The curves of Figs. 12 and 13 are not carried to fracture but, in general, are carried to beyond the upper yield point.

Curves of Fig. 14 are for tests at constant rate of load. These curves are carried to the breaking point. There is a rather marked difference in behavior in the material tested at constant rate of load as contrasted with constant rate of crosshead motion or constant rate of strain. As the material approaches its yield point, the machine speeds up to its maximum rate of 28 in. per min. in an attempt to maintain a constant rate of load increase. Even at this speed of crosshead motion, however, it is not possible in general to maintain a constant rate of load as the material passes through its yield point. However, the rate of increase is so great that the material, whether hard, soft, or medium, generally breaks. At this stage, instead of permitting the material to pass through the usual upper yield, lower yield and gradually increasing stress, the sudden increase in rate of strain occasioned by the attempt to maintain a constant rate of load does not give the material an opportunity to flow normally. The stress - strain curves in general appear to be curved right down to the origin compared to the straight-line behavior near the origin found in tests at constant rates of crosshead motion and at constant rates of ordinary strain. Generally speaking, the moduli of elasticity increase with increasing rates of load.

---

[4] Tentative Method of Test for Tensile Properties of Plastics (D 638 - 46 T), 1946 Book of ASTM Standards, Part III-B, p. 899.

The curves of tests run at constant rates of ordinary strain (Fig. 15) are similar to the curves of tests run at constant rates of crosshead motion (Figs. 12 and 13). Because the gage length of the extensometer is 1 in. whereas the distance between crosshead jaws is about 3 in., an initial ordinary strain rate of 0.450 in. per inch per min., for instance, is roughly comparable to an initial crosshead rate of approximately 1.28 in. per min. In cases where the strain rate is kept constant rather than the crosshead rate, as in Fig. 15, the curves are straight for a greater distance than are the curves of, say, Figs. 12 and 13. The maximum values at the upper yield point for the constant strain rate tests are also higher than are the values given in Fig. 13. The maximum stress values of curves 15 and 9 in Figs. 12, 13, and 15 are approximately 6600, 7100, and 7700 psi. for the different conditions indicated. Because the ordinary stress of Fig. 12 and the true stresses of Fig. 13 are taken from the same data, the increase from 6600 to 7100 psi. follows from the computations. The increase to 7700 psi. in Fig. 15, however, appears to follow from the conditions of the test, that is, constant rate of ordinary strain rather than constant rate of crosshead motion.

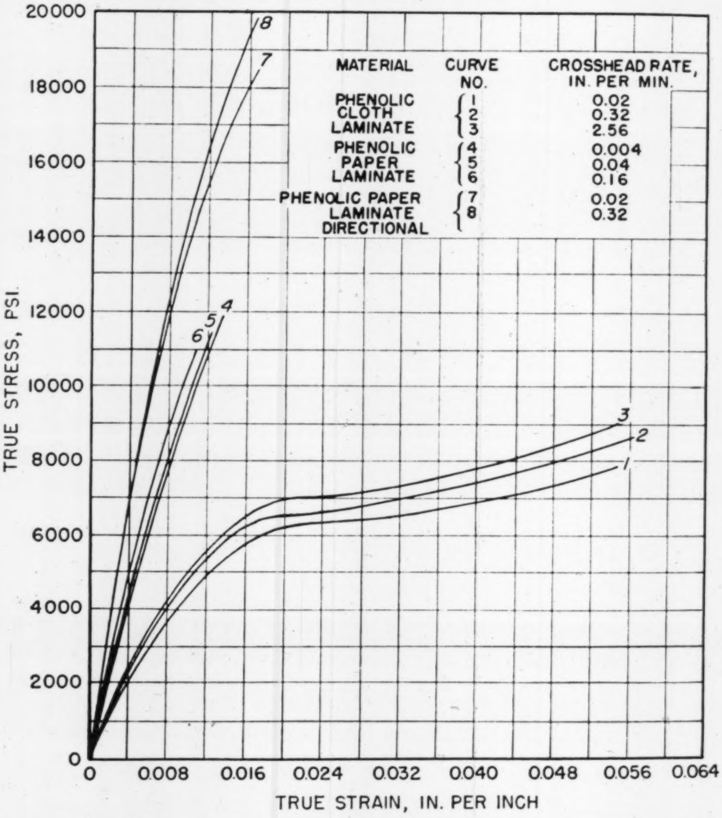


FIG. 16.—True Strain - True Stress Curves in Tension; Tests at Constant Rate Crosshead.

Paper and Fabric Laminates:

Tension tests were made of three laminates based upon phenolic resins. One was a non-directional paper laminate bearing the manufacturer's designation ER-6044, one a directional paper laminate CB-1107-1, and one a cotton fabric based laminate bearing the designation ER-6045.

Tests were carried out at constant rates of crosshead motion, constant rates of load, and constant rates of ordinary strain, with results shown in Figs. 16, 17, and 18. These are plots of true stress *versus* true strain. Because the strains are all small, the ordinary stress - ordinary strain plots would vary only slightly from these, so it was not deemed necessary to show both types of plots.

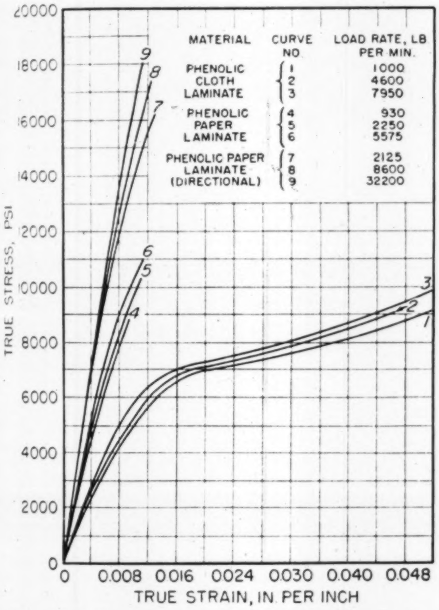


FIG. 17.—True Stress - True Strain Curves in Tension; Tests at Constant Rate of Load.

All three laminates show increasing values of maximum stress with increasing speed of test. Curves for the two types of paper laminates are similar for all three types of tests, and for the directional paper fall in the same general part of the graph. For the non-directional paper the tests at constant rates of ordinary strain are somewhat higher, that is, they show somewhat higher stresses for given strains than do the tests at constant rates of crosshead motion and constant rates of load. Because gage lengths were 1 in. and jaws of grips were approximately 3 in. apart, a test at constant rate of crosshead motion of 0.03 in. per min., for example, would correspond to an initial strain rate of approximately 0.01 in. per inch per min. Beyond this, comparisons are difficult because of such factors as slip in the jaws and variable extension of the wide and narrow portions of the test specimens (A.S.T.M. method D 638 - 46 T). Comparisons with constant rates of load are also difficult because of variable strain rates.

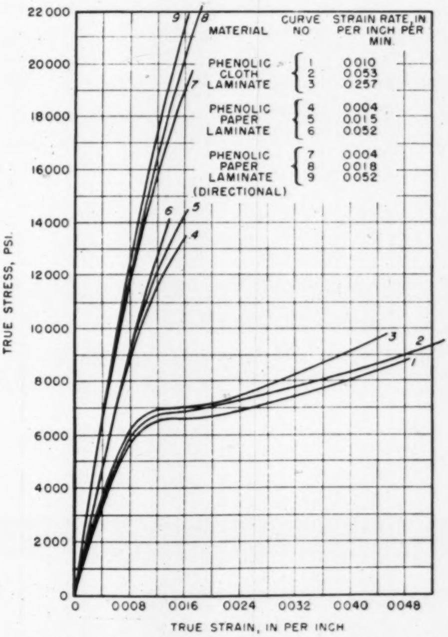


FIG. 18.—True Stress - True Strain Curves in Tension; Tests at Constant Rates of Strai

The fabric-based laminate showed

considerably larger extensions than the paper-based, and the stress-strain curves show a rather marked yield followed by a point of inflexion. It is believed that the "yield" shown in these curves might best be attributed to partial internal failures of the specimens causing discontinuities which could not be measured and thereby taken into account.

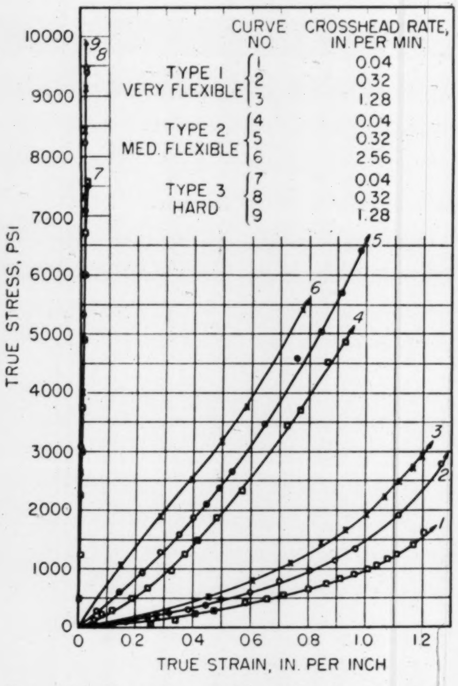


FIG. 19.—True Stress - True Strain Curves in Tension; Polyvinyl Chloride Types 1, 2 and 3; Tests at Various Constant Crosshead Rates.

In general, the sets of curves for tests at constant rates of load and constant rates of ordinary strain fall in the same general portion of the graph, and both are somewhat higher for a given strain than the curves of tests at constant rates of crosshead motion. Such comparisons can only be very general, however, because of the different conditions of the tests, especially the tests at constant rates of load. In this connection, it may be noted that the yielding of the fabric-based laminate was not so marked but that the machine was able to increase its speed sufficiently to maintain a constant rate of load. In this it differed from the cellulose acetate butyrate. Furthermore, unlike the cellulose acetate butyrate, failure did not occur at this point in tests at constant rate of load (compare Figs. 14 and 17).

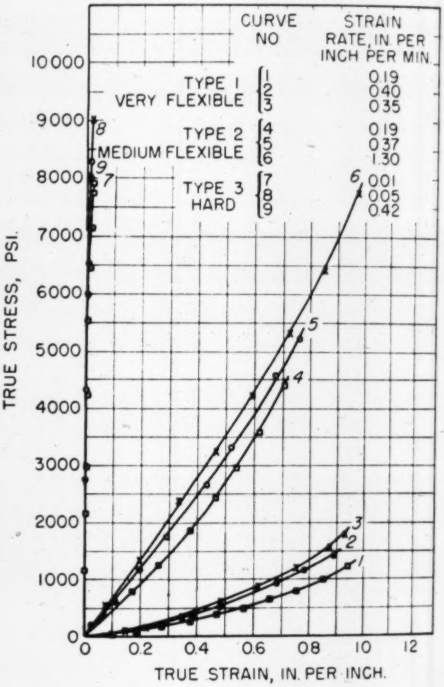


FIG. 20.—True Stress - True Strain Curves in Tension; Polyvinyl Chloride Types 1, 2 and 3; Tests at Various Constant Rates of True Strain.

Polyvinyl Chloride:

Three types of polyvinyl chloride were tested in tension. These represented widely different degrees of flexibility: relatively hard and rigid, moderately flexible, and very flexible and rubbery.

Two types of tests are reported here: constant rates of crosshead motion and constant rates of true strain. The true stress - true strain curves for constant rate of crosshead motion are shown in Fig. 19, true stress-true strain curve for constant rate of true strain in Fig. 20, and the same results re-plotted as ordinary stress - ordinary strain in Fig. 21. All of these curves show markedly the influence of speed of test. In every instance an increase in rate results in a substantial increase in stress for a given strain.

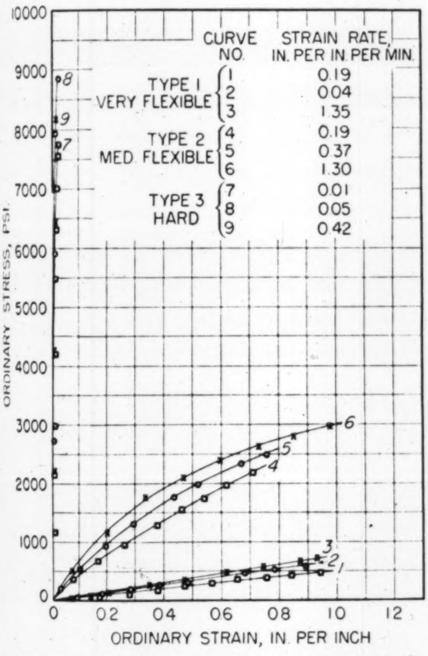


FIG. 21.—Ordinary Stress - Ordinary Strain Curves in Tension; Polyvinyl Chloride Types 1, 2 and 3; Tests at Various Constant Rates of True Strain.

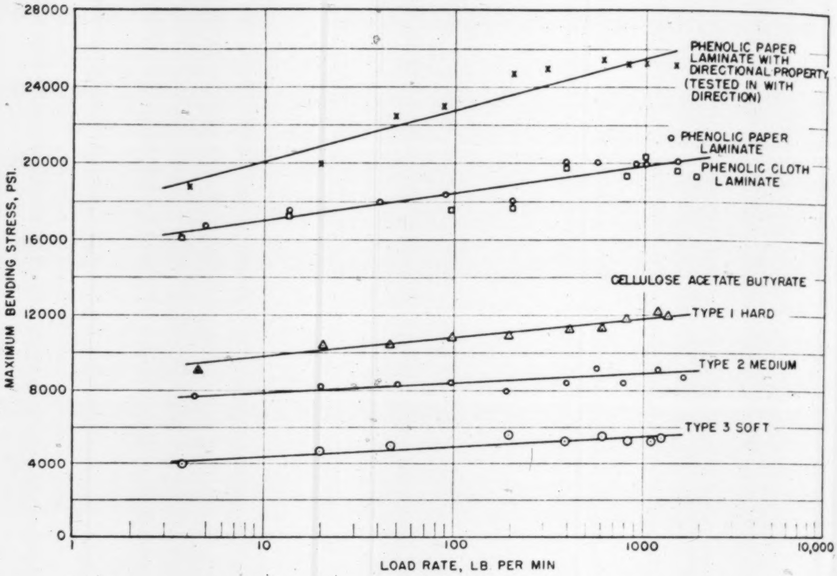
A comparison of Figs. 19 and 20 shows that tests at constant rates of true strain result in curves which lie generally above those run at constant rates of crosshead motion. The difference between the two types of test is very noticeable, especially with the moderately and highly flexible materials. Whereas in tests at constant rates of crosshead motion the crosshead moves at uniform speed and therefore at a decreasing rate of true strain (as well as ordinary strain because of slip in the jaws and variable extension of the specimen), at constant rate of true strain the crosshead moves at an accelerating rate and reaches a rapid pace toward the end of the tests. Therefore, no direct comparison can be made between the curves of Figs. 19 and 20.

The curves of Figs. 20 and 21 show the marked difference between true stress - true strain and ordinary stress - ordinary strain for extensible materials, like moderately and highly flexible polyvinyl chloride. These plots are calculated from the same test data. Not only do the curves lie in different parts of the graph, but the characters of the curves are different. As contrasted with Fig. 20, curves 1, 2, and 3 in Fig. 21 are virtually straight lines, and curves 4, 5 and 6 are increasingly concave downward with increasing strain rates. Since the tests represented by Fig. 21 were run at constant rates of true strain, these curves are the equivalent of tests run at a variable and increasing rate of ordinary strain. It would be expected, therefore, that tests run at constant rates of ordinary strain would result in curves still different in shape and more nearly like those of Fig. 19.

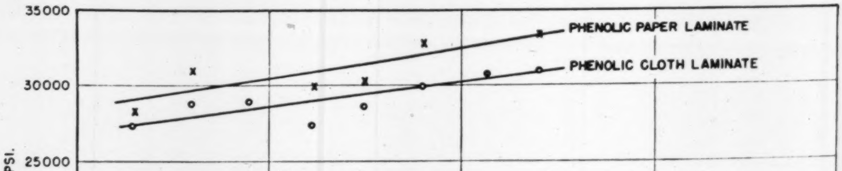
Ultimate Strengths in Bending and Compression:

Flexure tests were run at constant rates of load to determine the effect of varying rates upon the flexural strength of all three types of cellulose acetate butyrate, both types of paper laminate, and the fabric laminate. Compression tests were also run at constant rates of crosshead motion on the same materials plus a cast phenol formaldehyde. Flexure specimens were $\frac{1}{2}$ in. wide and $\frac{1}{4}$ in. thick (except directional paper which was $\frac{1}{8}$ in. thick), and were centrally loaded on a 4-in. span. Compression specimens were the same cross-sections and $\frac{3}{4}$ in. high.

Rates of load, varying from 4 to 1500



(a) Effect of Load Rate on Maximum Bending Stress.



(b) Effect of Crosshead Speed on Maximum Compressive Stress.

FIG. 22.

b. per min. and the resulting maximum bending stresses (moduli of rupture) are shown in Fig. 22(*a*). Rates of crosshead motion, varying from 0.02 to 28 in. per min., and the resulting maximum compressive stresses are shown in Fig. 22(*b*). It will be seen that in these materials the flexural maximum stresses are substantially linear functions of the logarithm of the rate of load, and the maximum compressive stresses are similar functions of the rate of crosshead motion. This confirms the tensile trends shown in Fig. 7 but not the findings of Lawton, Carswell, and Nason, for cellulose nitrate.

Polymethyl Methacrylate:

The relationship between tests at constant rates of crosshead motion and the true stress - true strain curves was studied by making tension and compression tests on a commercial polymethyl methacrylate. Tension specimens were $\frac{1}{4}$ in. thick and conformed in their shape to A.S.T.M. recommendations (D 638 – 46 T). Compression specimens were 0.353 by 0.353 by 0.750 in. As shown in Fig. 23, compression tests were run at rates of table travel ranging from 0.0037 to 2.25 in. per min. and tension tests at 0.0254 and 0.688 in. per min. Compressive strains were obtained by measuring the motion of the crosshead of the machine. Tensile strains were measured with SR-4 strain gages mounted with a special cement developed for polymethyl methacrylate (**21**). The resulting load-extension curves were recalculated by means of Eqs. 10 and 11 and the true stress - true strain curves of Fig. 23 were obtained. The relationship between the rate of table travel and the rate of true strain was obtained by plotting true strain against time (which was directly related to the motion of the table) as shown in Figs. 24 and 25. These are the same except for the horizontal scale which has been changed to bring out the nature of the relationship, particularly in curves 1 and 4.

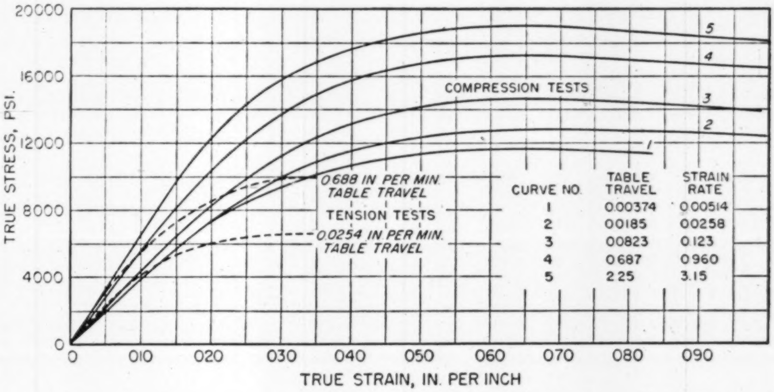
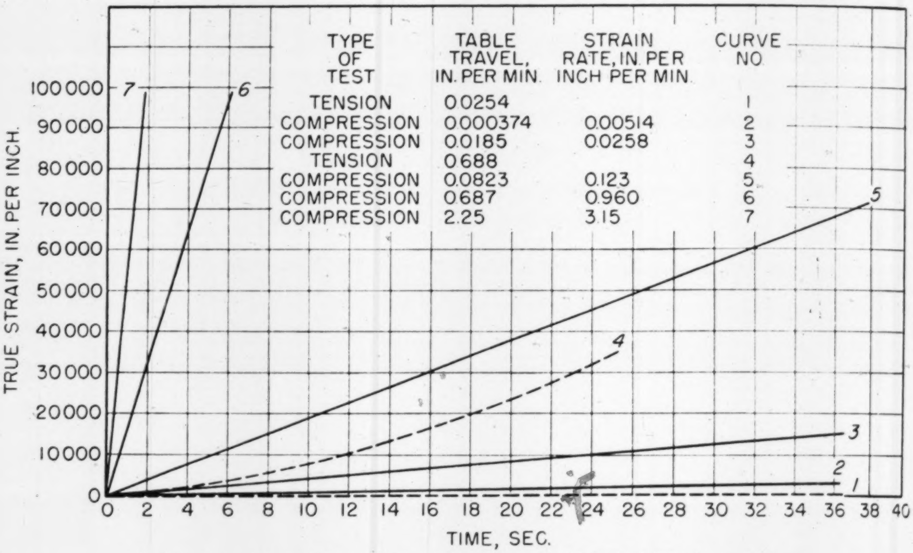


FIG. 23.—**True Stress - True Strain Curves., Commercial Methyl Methacrylate.**

It was found that in the compression tests a linear relationship existed between time and true strain (Figs. 24 and 25). This meant that compression tests run at constant rates of table travel could be interpreted in terms of constant rates of true strain. The corresponding rates are shown in Figs. 23, 24, and 25. The rate of true strain is approximately 35 to 50 per cent greater than the rate of table travel.

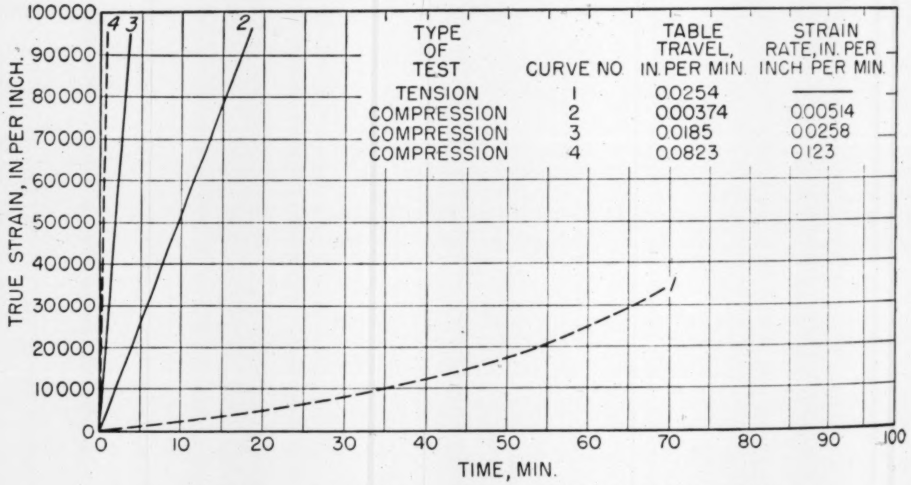
In tension tests, no linear relationship between true strain and time was found.

Consequently, rate of crosshead motion could not be interpreted in terms of a true strain rate since the strain rate changed as the tests progressed. This emphasized the importance of being able to control the rate of strain in a tension test, rather than the rate of crosshead motion, if a true picture of the stress-strain behavior of a material such as this was to be had.



TYPE OF TEST	TABLE TRAVEL, IN. PER MIN.	STRAIN RATE, IN. PER INCH PER MIN.	CURVE NO.
TENSION	0.0254		1
COMPRESSION	0.000374	0.00514	2
COMPRESSION	0.0185	0.0258	3
TENSION	0.688		4
COMPRESSION	0.0823	0.123	5
COMPRESSION	0.687	0.960	6
COMPRESSION	2.25	3.15	7

FIG. 24.—Rate of Strain Curve, Commercial Methyl Methacrylate.



TYPE OF TEST	CURVE NO	TABLE TRAVEL, IN. PER MIN.	STRAIN RATE, IN. PER INCH PER MIN.
TENSION	1	00254	———
COMPRESSION	2	000374	000514
COMPRESSION	3	00185	00258
COMPRESSION	4	00823	0123

FIG. 25.—Rate of Strain Curve, Commercial Methyl Methacrylate.

In order to obtain an insight into the true stress - true strain rates at various strains, plots (Fig. 26) were made of true stress *versus* true strain. In compression two curves were plotted, one for a true compressive strain $\epsilon_t = 0.025$ and one for $\epsilon_t = 0.065$. A single plot in tension for $\epsilon_t = 0.025$ was made. The two compression curves appear to be parabolic functions and may follow the power function proposed by Nadai (72):

$$s = f(u) = c(\bar{u})^m \qquad (14)$$

where:

u = the true strain rate, and
m and c = constants.

The tension curve is doubtful because it is plotted from only two points: also, the non-linearity of the true rates of strain in the tension tests makes it difficult to plot a true, stress - true strain rate curve.

An examination of Fig. 23 shows that in both the compression and tension tests the effect of increasing the cross-head motion was to raise both the elastic modulus and the maximum stress.

Like the other true stress - true strain curves reported above, the compression curves of Fig. 23 reach a maximum and then decrease. This again is contrary to theory and to experience with materials other than plastics and makes these curves open to question. Although the tension curves do not drop off before failure, the fact that they have levelled off makes them questionable. Internal failures, slight buckling, or the extreme difficulty of making good tests on rectangular specimens may all be involved.

A fundamental point respecting standard types of test specimens and the results obtained with them, particularly beyond the "yield points" and at large extensions, may be involved and needs further clarification.

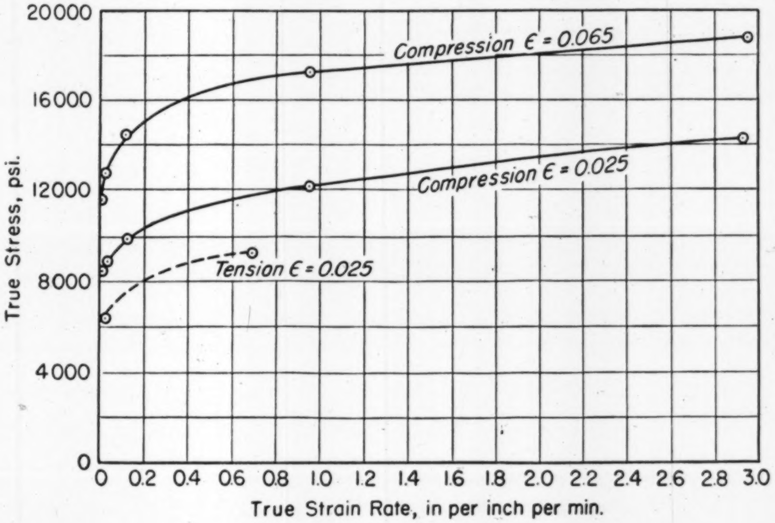


FIG. 26.—True Stress - True Strain Rate Curves, Commercial Methyl Methacrylate in Compression and Tension.

Stress-Strain-Time Relationships:

G. Murphy (70) and others have proposed and discussed the advantages and characteristics of three-dimensional diagrams of stress, strain, and time. In such diagrams curved surfaces are obtained, and sections taken through these surfaces parallel to the stress-strain, stress-time, and strain-time planes could be used to portray the behavior of the material under various loading conditions.

The compression curves of Figs. 23,

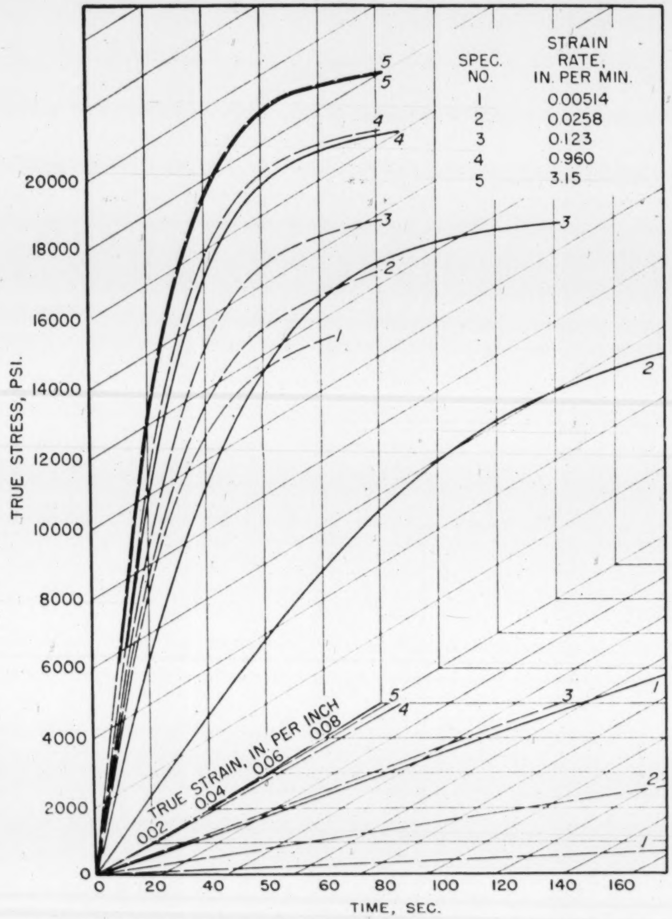


FIG. 27.—True Stress - True Strain Time Relations. Polymethyl Methacrylate in Compression.

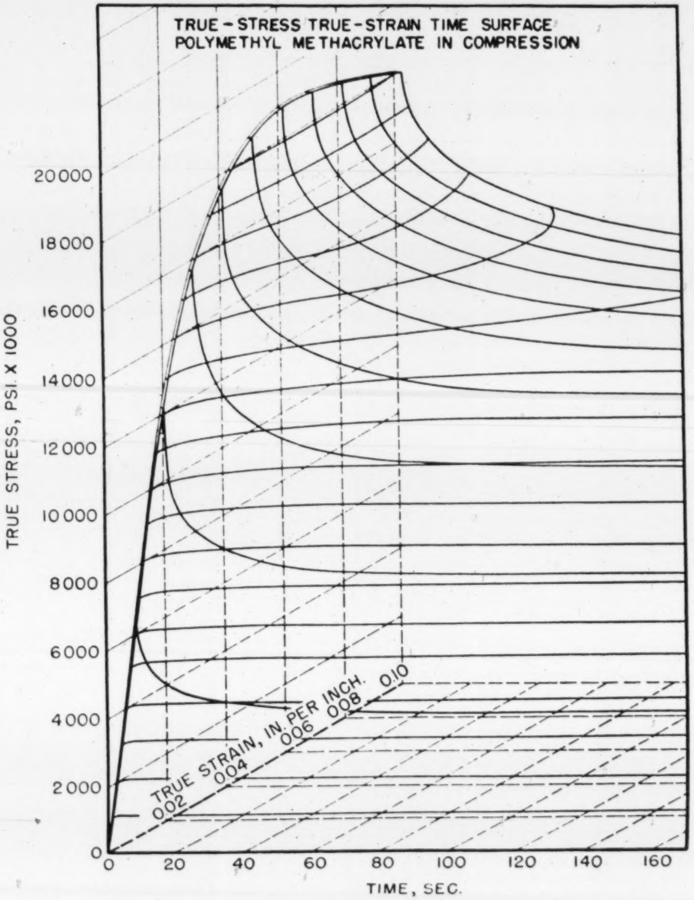


FIG. 28.—True Stress - True Strain Time Surface. Polymethyl Methacrylate in Compression.

24, and 25 were accordingly combined to provide the three-dimensional compression diagrams shown in Figs. 27 and 28. Figure 27 shows the method of development of the three-dimensional plot. On the horizontal, or true strain-time plane, the compression strain rate curves of Figs. 24 and 25 were plotted and upon these were erected the corresponding true stress-time-strain curves of Fig. 23. The solid lines on Fig. 27 are the curves of Fig. 23, the dotted lines are projections on the stress-strain plane. These gave the three-dimensional surface. It will be seen that a substantial part of the entire volume is provided by these curves and that only the very high and very low rate portions of the surface are missing. Within the limits of these surfaces, contours can be taken parallel to the three planes of reference or at any desired angle to depict the true stress - true strain - time behavior. The contours drawn parallel to the time-stress plane correspond to relaxation conditions (decreasing stress at constant strain) and the contours drawn parallel to the time-strain plane correspond to creep conditions (increasing strain at constant stress). Relaxation contours are typical of relaxation tests in which the stress decays at a decreasing rate. As they approach the zero time plane they rise sharply.

The creep contours appear to possess certain characteristics due to the fact that the three-dimensional surface rises to a maximum stress value and then drops off. As has been pointed out, this is questionable. As a consequence, the creep curves (horizontal contours) appear to reach a maximum time value and then curve back. Since this is a physical anomaly, it might be postulated that in actual creep tests failure, that is, a rapid increase in compression, would occur when time had reached the maximum point on the curve. However, the arguments advanced above indicate that the entire surface beyond the maximum stress point is open to question. Before any further interpretation of these contours can be attempted, more precise studies of tension, compression, relaxation, and creep tests will be required to check the validity of the depicted behavior on the true stress - true strain curve.

Acknowledgments:

The tests reported here are a part of the program of fundamental research into the mechanical properties of plastics sponsored at M.I.T. by the Plastic Materials Manufacturers Association. Particular credit is due to Mrs. Doris Cleveland, who worked hard on the stress-strain data, and to H. G. Lauterbach, Earl Patterson, W. H. Campbell and R. H. Kimble.

SELECTED BIBLIOGRAPHY

(1) A. P. Alaksandrov and IU. S. Lazurkin "Study of Polymers. I. Highly Elastic Deformation in Polymers," *Acta Physicochim. U.R.S.S.*, Vol. 12, pp. 647–668 (1940).

(2) T. Alfrey, "A Molecular Theory of the Viscoelastic Behavior of an Amorphous Linear Polymer," *Journal of Chemical Physics*, Vol. 12, pp. 374–379 (1944).

(3) T. Alfrey, Jr., "Mechanical Behavior of High Polymers," Interscience Publishers, Inc., New York, N. Y. (1948).

(4) Amsterdam, University of, "First Report on Viscosity and Plasticity," Amsterdam (1935).

(5) W. F. Bartoe, "Service-Temperature Flow Characteristics of Thermoplastics," *Mechanical Engineering*, Vol. 61, No. 12, December, 1939, pp. 892–894.

(6) W. F. Bartoe, "Strength and Properties of Plexiglass," *Aviation*, Vol. 42, January, 1943, pp. 128–131, 135.

(7) W. F. Bartoe and D. S. Frederick, "Characteristics of Plastics as Engineering Materials," *Transactions*, Soc. Automotive Engrs., Vol. 52, No. 2, February, 1944.

(8) L. Boltzmann, "Zur Theorie der elastichen Nachwirkung," Sitzber. Kgl. Akad. Wiss. Wien, Math.-Naturw. Classe, *70*, p. 275 (1874) = Pogg. Ann. Physik, *7*, 624 (1876).

(9) L. Boltzmann, "Zur Theorie der elastichen Nachwirkung," *Wied. Ann. Physik*, Vol. *5*, p. 430 (1878).

(10) S. E. Bresler and IA. I. Frenkel, "On the Shape and Character of Thermal Motion of Long-Chain Organic Molecules and on the Elasticity of Rubber-like Substances," *Acta Physicochim.* U.R.S.S., Vol. *11*, p. 485–504 (1939).

(11) Robert Burns and W. W. Werring, "The Impact Testing of Plastics," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 39 (1938).

(12) G. S. Burr, W. J. Gailus, J. O. Silvey, S. Yurenka, A. G. H. Dietz, "Universal Plastics Testing Machine," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 1030 (1947); ASTM BULLETIN, No. 149, December, 1947, p. 51.

(13) Harris Burte and George Halsey, "A New Theory of Non-Linear Viscous Elasticity," *Textile Research Journal*, Vol. XVII, No. 9, September, 1947.

(14) L. H. Callender, "New Methods for Mechanical Testing of Plastics," Part I, *British Plastics*, Vol. *13*, No. 155, April, 1942, pp. 445–458.

(15) W. H. Carothers, "Collected Papers on High Polymeric Substances" (ed. H. Mark, G. S. Whitby), High Polymers, Vol. 1, New York, N. Y. (1940).

(16) T. S. Carswell, R. F. Hayes, and H. K. Nason, "Physical Properties of Polystyrene as Influenced by Temperature," *Industrial and Engineering Chemistry*, Vol. *34*, p. 454 (1942).

(17) T. S. Carswell and H. K. Nason, "Effect of Environmental Conditions on the Mechanical Properties of Organic Plastics," *Symposium on Plastics*, Am. Soc. Testing Mats., p. 22 (1944).

(18) John Delmonte, "Elastic Properties of Plastic Materials," *Transactions*, Am. Soc. Mechanical Engrs., Vol. *67*, pp. 477–481 (1945).

(19) H. de Witt Smith and R. Eisenschitz, "Flow and Relaxation of Rayon Filaments," *Journal of the Textile Institute*, Vol. *22*, p. T170 (1931).

(20) G. J. Dienes, "Viscoelastic Properties of Thermoplastics at Elevated Temperatures," *Journal of Colloid Science*, Vol. 2, No. 1, February, 1944, pp. 131–161.

(21) A. G. H. Dietz and W. H. Campbell, "Bonded Wire Strain Gage Techniques for Polymethyl Methacrylate Plastics," *Proceedings*, Soc. for Exp. Stress Anal., Vol. 5, No. 1 (1947).

(22) W. N. Findley, "Fatigue Tests of a Laminated Mitscherlich-Paper Plastic," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 878 (1945).

(23) W. N. Findley, "Mechanical Tests of Macerated Phenolic Molding Material," National Advisory Committee for Aeronautics, Wartime Report W 99.

(24) W. N. Findley, "Mechanical Tests of Cellulose Acetate," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1231 (1941); Vol. 42, p. 914 (1942).

(25) W. N. Findley, "Mechanical Tests of Cellulose Acetate," *Modern Plastics*, Vol. 20, No. 7, March, 1943, pp. 99–105, 138.

(26) Paul J. Flory, "Effects of Molecular Structure on Physical Properties of Butyl Rubber," *Industrial and Engineering Chemistry*, Vol. *38*, No. 4, 417–36 (1946).

(27) P. J. Flory and J. Rehner, "Statistical Theory of Chain Configuration and Physical Properties of High Polymers. Effects of Hindered Rotation," New York Academy of Sciences Conference on High Polymers, January 9, 1943.

(28) P. J. Flory, "Viscosities of Linear Polyesters. An Exact Relationship Between Viscosity and Chain Length," *Journal*, Am. Chem. Soc., Vol. *62*, p. 1057 (1940).

(29) J. Frenkel and J. Obrastov, "A Phenomenological Theory of the Mechanical Properties of Amorphous Bodies," *Journal Physics* (U.S.S.R.), Vol. *2*, p. 131 (1940).

(30) W. J. Gailus and David Telfair, "Creep Properties of Molded Phenolic Plastics at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. *67*, pp. 253–258 (1945).

(31) S. Glasstone, K. J. Laidler, and H. Eyring, "The Theory of Rate Processes," Chapter IV, McGraw-Hill Book Co., Inc., New York, N. Y. (1941).

(32) G. Gurevitch and P. Kobeko, "On the Technique of the Mechanical Testing of Soft and Hard Rubber and Plastics," *Acta Physicochim. U.R.S.S.*, Vol. *12*, p. 681 (1940).

(33) E. Guth, "The Physics of Rubber. I. Its Observed Elastic and Thermoelastic Behavior," *Journal of Applied Physics*, Vol. *10*, p. 201 (1939).

(34) E. Guth and H. James, "Elastic and Thermoelastic Properties of Rubber-like Materials. A Statistical Theory," *Industrial and Engineering Chemistry*, Vol. *33*, pp. 624–629 (1941).

(35) George Halsey, "Non-linear Viscous Elasticity and the Eyring Shear Model," *Journal of Applied Physics*, Vol. *18*, No. 12, December, 1947, p. 1072.

(36) R. N. Haward, "The Extension and Rupture of Cellulose Acetate and Celluloid," *Transactions*, Faraday Soc., Vol. *38*, pp. 394–403 (1942).

(37) R. N. Haward, "The Fast and Slow Extension of Some Plastic Materials," *Transactions*, Faraday Soc., Vol. *39*, Part II, No. 254, November, 1943, pp. 267–280.

(38) J. Hopkinson, "On the Torsional Strain which Remains in a Glass Fibre after Release from Twisting Stress," *Proceedings*, Royal Soc. (London), Vol. *28*, p. 148 (1878).

(39) R. Houwink, "Elasticity, Plasticity and the Structure of Matter," Cambridge University Press, London (1937).

(40) R. Houwink, "High Elasticity of Three Dimensionally Polymerised Amorphous Materials in Relation to Their Structure," *Transactions*, Faraday Soc., Vol. *32*, p. 131, (1936).

(41) H. James and E. Guth, "Theory of the Elasticity of Rubber," *Journal Applied Physics*, Vol. *15*, No. 4, April, 1944, pp. 294–303.

(42) H. James and E. Guth, "Theory of Rubber Elasticity for Development of Synthetic Rubbers," *Industrial and Engineering Chemistry*, Vol. *34*, p. 1365 (1942).

(43) H. James and E. Guth, "Theoretical Stress-Strain Curve for Rubber-like Materials," *Physical Review*, Vol. *59*, p. 111 (1941).

(44) H. Jeffreys, "On Plasticity and Creep in Solids," *Proceedings*, Roy. Soc. (London) A., Vol. *138*, p. 283 (1932).

(45) W. J. Kauzmann and H. Eyring, "The Viscous Flow of Large Molecules," *Journal*, Am. Chemical Soc., Vol. *62*, p. 3113 (1940).

(46) F. Kohlrausch, "Beiträge zur Kenntniss der elastichen Nachwirkung," *Pogg. Ann. Physik*, (5) Vol. *8*, No. 1, pp. 207, 399 (1866).

(47) F. Kohlrausch, "Experimental-Untersuchung über die elastiche Nachwirkung bei der Torsion, Ausdehnung, und Biegung," *Pogg. Ann. Physik*, (6) Vol. *8*, p. 337 (1876).

(48) F. Kohlrausch, "Ueber die elastiche Nachwirkung bei der Torsion," *Pogg. Ann. Physik*, (4) Vol. *29*, p. 337 (1863).

(49) R. Kohlrausch, "Nachtrag über die elastiche Nachwirkung beim Cocon und Glasfaden, etc.," *Pogg. Ann. Physik*, (3) Vol. *12*, p. 393 (1847).

(50) W. Kuhn, "Beziehungen zwischen Viscosität und elastichen Eigenschaften Amorpher Stoffe," *Z. Physik. Chem.*, Vol. B42, pp. 1–38 (1939).

(51) T. S. Lawton, Jr., T. S. Carswell, and H. K. Nason, "Effect of Some Environmental Conditions on the Mechanical Properties of Cellulose Acetate and Cellulose Nitrate Plastic Sheets," *Transactions*, Am. Soc. Mechanical Engrs., Vol. *67*, January, 1945, pp. 23–30.

(52) H. Leaderman, "Creep and Creep Recovery in Plasticized Polyvinyl Chloride," *Industrial and Engineering Chemistry* (Ind. Ed.), Vol. *35*, p. 374 (1943).

(53) H. Leaderman, "Creep, Elastic Hysteresis, and Damping in Bakelite under Torsion," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 61, A-79 (1939).

(54) H. Leaderman, "Elastic and Creep Properties of Filamentous Materials and Other High Polymers," The Textile Foundation, Washington, D. C.

(55) H. Leaderman, "Mechanical Behavior of Textile Fibers and Plastics," *Textile Research*, Vol. *11*, p. 171 (1941).

(56) H. Leaderman, "Textile Materials and the Time Factor. I. Mechanical Behavior of Textile Fibers and Plastics," *Textile Research*, Vol. *11*, pp. 171–193 (1941).

(57) P. Ludwik, "Elemente der Technologischen Mechanik," Julius Springer, Berlin (1909).

(58) C. W. MacGregor, "The Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 508 (1940).

(59) C. W. MacGregor, "The True Stress-Strain Tension Test—Its Role in Modern Materials Testing," Parts I and II, *Journal of the Franklin Institute*, Vol. 238, Nos. 2 and 3, August and September, 1944, pp. 111–135 and 159–176.

(60) J. Marin, "Tension and Torsion Creep Properties of Cloth Laminates," ASTM Bulletin, No. 143, December, 1946, p. 38.

(61) H. Mark, "Fine Structure and Mechanical Properties of Fibres," *Transactions*, Faraday Soc., Vol. *29*, p. 6 (1933).

(62) H. Mark, "Intermolecular Forces and Mechanical Behavior of High Polymers," *Industrial and Engineering Chemistry*, Vol. *34*, p. 1343 (1942).

(63) H. Mark, "Natural and Synthetic Rubber. The Elasticity of Long-Chain Molecules," *Nature*, Vol. *141*, pp. 670–672 (1938).

(64) H. Mark, "Quantitative Ansätze zur Enfassung der Formänderung hochpolymerer Stoffe," Congr. intern. quim. pura aplicada, 9th Congr., Madrid, Vol. *4*, pp. 197–207 (1934).

(65) H. Mark, "Structure and Mechanical Behavior of High Polymers," *Cold Spring Harbor Symposia on Quantitative Biology*, Vol. *9*, pp. 204–209 (1941).

(66) J. Clerk Maxwell, "Constitution of Bodies," *Encyclopaedia Britannica*, 6th Edition, Vol. *9*, p. 315 (1877); *Collected Works*, Vol. *2*, p. 616, Cambridge (1890).

(67) J. Clerk Maxwell, "The Dynamical Theory of Gases," Phil. *Transactions*, Roy. Soc. (London), Vol. *157*, p. 52 (1867); *Scientific Papers*, Vol. *2*, p. 26, Cambridge, 1890.

(68) J. Miklowitz, "The Initiation and Propagation of the Plastic Zone Along a Tension Specimen of Nylon," *Journal of Colloid Science*, Vol. *2*, No. 1, February, 1947, pp. 193–215.

(69) D. R. Morey, "Impact Testing of Plastics—Relation to Structure and Composition," *Industrial and Engineering Chemistry*, Vol. *37*, March, 1945, p. 255.

(70) Glenn Murphy, "Stress-Strain-Time Characteristics of Materials," ASTM BULLETIN, No. 101, December, 1939, p. 19.

(71) C. S. Myers, "Impact Strength of Plastic Sheet," *Modern Plastics*, October (1942).

(72) A. Nadai and M. J. Manjoine, "High Speed Tension Tests at Elevated Temperatures," *Journal of Applied Mechanics*, June, 1941, pp. A77–A91.

(73) A. Nadai, "Plasticity," McGraw-Hill Book Co. Inc., New York, N. Y., pp. 80–85 (1931).

(74) W. Prager, "Introduction to the Mathematical Theory of Plasticity," *Journal of Applied Physics*, Vol. *18*, pp. 375–83 (1947).

(75) R. K. Schofield and G. W. Scott-Blair, "The Relationship between Viscosity, Elasticity and Plastic Strength of Soft Materials, etc.," *Proceedings*, Roy. Soc. (London) A., Vol. *138*, p. 707 (1932).

(76) R. Simha, "Anomalies of Elasticity and Flow and Their Interpretation," *Journal of Physical Chemistry*, Vol. *47*, pp. 348–63 (1943).

(77) R. Simha, "On Relaxation Effects in Amorphous Media," *Journal of Applied Physics*, Vol. *13*, p. 201 (1942).

(78) D. Telfair, C. H. Adams, and H. W. Mohrman, "Creep, Long-time Tensile and Flexural Fatigue Properties of Melamine and Phenolic Plastics Materials," *Transactions*, Am. Soc. Mechanical Engrs., Vol. *69*, pp. 789–93 (1947).

(79) D. Telfair and H. K. Nason, "Impact Testing of Plastics. I. Energy Considerations," *Modern Plastics*, Vol. 20, No. 11, p. 85 (1943).

(80) D. Telfair and H. K. Nason, "Impact Testing of Plastics. II. Factors which Influence the Energy Absorbed by the Specimen," *Modern Plastics*, April, 1945, pp. 145–49, 186.

(81) J. H. C. Thompson, "On the Theory of Viscoelasticity, etc.," Phil. *Transactions*, Roy. Soc. (London) A., Vol. *231*, p. 339 (1933).

(82) August Thum and Hans-Rudolf Jacobi, "Mechanical Strength of Phenol: Formaldehyde Plastics," *VDI—Forschungsheft 396* (Supplement to Forschung auf dem Gebiete des Ingenieurwesens Vol. *10B*, May/June, 1939.

(83) A. V. Tobolsky and R. D. Andrews, "Systems Manifesting Superposed Elastic and Viscous Behavior," *Journal of Chemical Physics*, Vol. *13*, No. 1, January, 1945, pp. 3–27.

(84) A. Tobolsky and H. Eyring, "Mechanical Properties of Polymeric Materials," *Journal of Chemical Physics*, Vol. *11*, p. 125 (1943).

(85) A. Tobolsky, R. E. Powell, and H. Eyring, "Elastic-Viscous Properties of Matter in *The Chemistry of Large Molecules* (*Frontiers in Chemistry*, Vol. I)," Interscience Publishing Co., New York, N. Y., 1943, Chapter V.

(86) R. F. Tuckett, "The Kinetics of High Elasticity in Synthetic Polymers," *Transactions*, Faraday Soc., Vol. *38*, pp. 310–322 (1942); *Rubber Chemistry and Technology*, Vol. *15*, pp. 430–7 (1942).

(87) R. F. Tuckett, "The Kinetics of High Elasticity in Synthetic Polymers," *Rubber Chemistry and Technology*, Vol. *16*, pp. 760–766 (1943).

(88) E. Wiechert, "Gesetze der elastichen Nachwirkung für constante Temperatur," *Wied. Ann. Physik.*, Vol. *50*, pp. 335, 546 (1893).

(89) W. Weber, "Ueber die Elasticität der Seidenfäden," *Pogg. Ann. Physik*, (2), Vol. *4*, p. 247 (1835).

(90) W. Weber, "Ueber die Elasticität fester Körper," *Pogg. Ann. Physik*, (2) Vol. *24*, p. 1 (1841).

(91) W. A. Zinzow, "Flexural Properties of Plastics," ASTM BULLETIN, No. 134, May 1945, p. 31.

DISCUSSION

Mr. H. K. Nason.[1]—It is very encouraging to note that the objective of this symposium has been stated as a "stimulation of further study and improvement." Certainly, in the case of plastics, our ignorance with respect to mechanical behavior is tremendous. Quantitatively, our information has been and still is pitifully inadequate.

I want to congratulate Mr. Dietz and his co-workers for directing their efforts towards the improvement of this situation, first, by devising improved apparatus which will enable us to cover a wider range of loading or deforming conditions (a necessity if we are to understand more completely the mechanical behavior of plastics) and, second, by applying this new equipment in basic studies of the rheological behavior of plastic materials.

This second paper in their series, reporting application of the new testing machine to commercial plastic materials, has revealed interesting information on their mechanical behavior. However, there still exists a great many blanks in our knowledge, as has been pointed out. Further, we recognize that we have a long way to go before we really understand the relationship that exists between laboratory determined physical properties and service or use behavior.

I feel confident that as this work goes on, we shall eventually reach the place where we can accurately predict the behavior of plastic materials. When that time comes, we shall be able to use them safely as engineering materials, in a great variety of applications, where we now can only cut and try (and rather expensively) because sound engineering bases for our original designs are lacking.

The authors have brought out some points bearing on the relationship between nominal stress and true stress and ordinary strain and true strain which will undoubtedly be of importance to our work in this field and to a long-range understanding of these materials.

A few years ago we thought we were doing pretty well when we were able to test plastics at constant rate of crosshead motion. It represents a real advance in our technique that we are now able to test at constant rate of either ordinary or "true" strain. The availability of new testing equipment and newer testing techniques affords us new possibilities. However, the relative merits of the various modes of operation have yet to be established. Certainly, we shall not be able to do so, reasonably, until we have applied these various new techniques to a wide range of plastic materials and under even a wider range of test conditions than the authors have so far been able to achieve. It should be emphasized that this is a problem that is going to require very serious attention not only on the part of those in the plastics field but also by those in some of the older materials' fields.

What type of testing technique will we eventually arrive at, or perhaps I should say, what types? Is true strain going to be the most informative data for us or will ordinary strain be adequate? Will we have to evaluate our results in terms of true stress, or will we be able to proceed on the basis of nominal stress, as we have in the past?

---

[1] Associate Director, Central Research Dept., Monsanto Chemical Co., Dayton, Ohio.

These considerations will require a great deal of further study by other workers in these fields.

MESSRS. A. G. H. DIETZ, W. J. GAILUS, and S. YURENKA (*authors*).—Mr. Nason is quite right in emphasizing that at this stage we are not at all certain what type of information is going to be most useful in plastics design. Until now older structural materials like steel have customarily been designed on the basis of ordinary stress-ordinary strain relationships. Such designs have been generally satisfactory as long as strains were small and the material was essentially elastic in its behavior. Even with these materials, however, some present-day applications involving plastic deformation require more exact analyses on the basis of true stress-true strain relationships. It seems logical, therefore, to suppose that the plastics which generally behave plastically rather than elastically will best be handled on the basis of true stress and true strain. Much more work will be required to decide this point.

MR. C. HOWARD ADAMS[2] (by letter).—The authors have done an excellent job in collecting and presenting the important information contained in this paper. The bibliography represents a thorough coverage of the field.

It is to the credit of the authors that they have recognized the limitations of ordinary stress-strain determinations. However, there is some question regarding the significance of the term "true strain." There are numerous ways to define strain and the reason for calling $ln \frac{l}{l_o}$ "true" strain is somewhat obscure. In terms of the components of the stress and strain tensors for large deformations, it is quite unlikely that any such simple relation as the "true" strain has much more significance than the ordinary strain. However, it certainly makes more sense to use "true" stress and "true" strain as defined here than to use the ordinary stress and strain as long as no satisfactory theory of large deformations exists. A good mathematical theory of large deformations is needed.

In some cases it would appear that elongation defined as

$$\frac{(\text{instantaneous length} - \text{initial length})}{(\text{instantaneous length})} = \frac{\Delta l}{l}$$

is a better relation than $ln \frac{l}{l_o}$. The two are nearly identical for moderate strains. The following reference is of considerable interest on the subject of finite strain:

F. D. Murnaghan, "Finite Deformations of an Elastic Solid," *Am. Journal Math.*, Vol. 59, pp. 235–260 (1937).

The following proof is presented merely as a matter of interest to those concerned with the mathematics and geometry of the "true" strain extensometer:

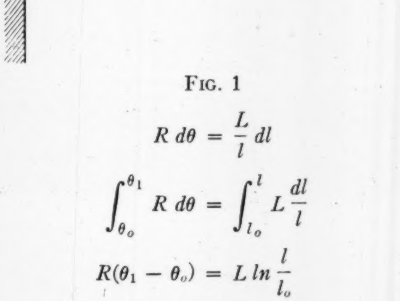


FIG. 1

$$R\, d\theta = \frac{L}{l}\, dl$$

$$\int_{\theta_o}^{\theta_1} R\, d\theta = \int_{l_o}^{l} L \frac{dl}{l}$$

$$R(\theta_1 - \theta_o) = L\, ln \frac{l}{l_o}$$

---

[2] Research Department, Plastics Div., Monsanto Chemical Co., Springfield, Mass.

By definition,

$$ln\,\frac{l}{l_o} = \text{“true” strain}$$

thus

$$\epsilon_t = ln\,\frac{l}{l_o} = \frac{R}{L}(\theta_1 - \theta_o) = \frac{R\theta}{L}$$

where:

L = distance from knife edge to axis of rotation of measuring wheel,
R = radius of measuring wheel,
θ = angular displacement of measuring wheel,
l_o = initial length,
l = final length,
dl = increment of elongation, and
ϵ_t = “true” strain.

TABLE I.

$\frac{L}{L_o}$	$ln\,\frac{L}{L_o}$	$\frac{L-L_o}{L}$	$\frac{L-L_o}{L_o}$	$\frac{L-L_o}{\frac{1}{2}(L+L_o)}$
1.001	0.0009995	0.0009990	0.001	0.0009995
1.010	0.00995	0.00990	0.010	0.00995
1.100	0.09531	0.0909	0.100	0.0952
1.200	0.1823	0.1668	0.200	0.182
1.300	0.2624	0.231	0.300	0.261
1.400	0.3365	0.286	0.400	0.334
1.500	0.4055	0.334	0.500	0.400
2.00	0.69315	0.500	1.000	0.666
5.00	1.6094	0.800	4.000	1.333
10.00	2.303	0.900	9.000	1.635

MESSRS. DIETZ, GAILUS, AND YURENKA.—Mr. Adams is quite right in pointing out that a good mathematical theory for larger deformations is still needed. In fact the development of a theory of large elastic deformations has not yet progressed towards practically applicable solutions. However, it still remains to be noted that the definition of strain used affects the variables used in describing the action of a finite body under the influence of forces unless the deformations are infinitesimal. If the strains are finite, the action of these forces must be related to the actual strained shape in order to obtain a true picture of what is going on. The definition of strain, which to date most conveniently represents this “true” picture, seems to be that suggested by Ludwik and Hencky:

$$\epsilon_t = \int_{L_o}^{L} \frac{dl}{l} = ln\,\frac{L}{L_o}.$$

If we expand $ln\,\frac{L}{L_o}$ in a series thus

$$ln\,\frac{L}{L_o} = \left[\frac{\left(\frac{L}{L_o} - 1\right)}{\frac{L}{L_o}}\right] + \frac{1}{2}\left[\frac{\frac{L}{L_o} - 1}{\frac{L}{L_o}}\right]^2 + \frac{1}{3}\left[\qquad\right]^3 + \cdots$$

and neglect all terms on the right beyond the first we have

$$ln\,\frac{L}{L_o} \approx \frac{L - L_o}{L} = \frac{\Delta L}{L}$$

which is seen to be an approximation to the original definition and good only to the point where the terms dropped in the series expansion are negligible. Another type of expansion

$$ln\,\frac{L}{L_o} = \left(\frac{L}{L_o} - 1\right) - \frac{1}{2}\left(\frac{L}{L_o} - 1\right)^2 + \frac{1}{3}\left(\frac{L}{L_o} - 1\right)^3 - \cdots$$

gives, upon neglecting all terms beyond the first

$$ln\,\frac{L}{L_o} \approx \frac{L - L_o}{L_o}$$

TABLE II.

$\frac{L}{L_o}$	$ln\,\frac{L}{L_o}$	$\frac{L-L_o}{L}$	$\frac{L-L_o}{L_o}$	$\frac{L-L_o}{\frac{1}{2}(L+L_o)}$
1.001	1.000	0.9995	1.0005	1.0000
1.010	1.000	0.9950	1.0050	1.0000
1.100	1.0000	0.9537	1.0492	0.9988
1.200	1.0000	0.9150	1.0971	0.9984
1.300	1.0000	0.8803	1.1433	0.9947
1.400	1.0000	0.8499	1.1887	0.9926
1.500	1.0000	0.8237	1.2330	0.9864
2.00	1.0000	0.7213	1.4427	0.9608
5.00	1.0000	0.4971	2.4854	0.8283
10.00	1.0000	0.3908	3.9079	0.7099

that is, the definition of ordinary strain. A third expansion

$$\ln\frac{L}{L_o} = 2\left\{\left[\frac{\dfrac{L}{L_o}-1}{\dfrac{L}{L_o}+1}\right] + \frac{1}{3}\left[\frac{\dfrac{L}{L_o}-1}{\dfrac{L}{L_o}+1}\right]^3 + \frac{1}{5}\left[\qquad\right]^5 + \cdots\right\}$$

upon similarly neglecting all terms beyond the first gives

$$\ln\frac{L}{L_o} \approx \frac{L - L_o}{\dfrac{L + L_o}{2}} = \frac{\Delta L}{\frac{1}{2}(L + L_o)}$$

another commonly used strain definition.

It is of interest to compare these various approximations to the original true strain definition for various strains. This has been done in the accompanying Table I. To bring out the divergence from the exact value in Table II, the exact value has been made equal to unity in each case. The implications are obvious. A very readable and important exposition on this question appears in the paper by C. W. MacGregor.[3]

MR. D. A. SHINN[4] (*by letter*).—This paper presents some very interesting and valuable data relative to the relation of rates of loading, crosshead speed, and straining and should be extended. However, it was noted that a rather unusual discrepancy occurred in the ratio of head travel to strain between tension and compression loading, that is, the ratio for varying stresses in tension was not the straight line which might be expected, but in compression it did become a straight line indicating that head travel was directly and linearly a function of strain in the specimen.

Since such a situation appears somewhat unusual, it is suggested that the method of compressive strain measurement may have been the cause. It was understood that the compressive strain gage measured strain or deformation between the *ends* of the compressive specimen. Such a test should be proportional to head travel, especially since one head is effectively stationary on this machine. In other words the test plotting seems to prove that $x = x$.

It is well known that such compressive strain measurements are considerably in error, such error for moduli determinations amounting to as much as 50 per cent for balsa and perhaps 10 per cent for aluminum alloys as compared to results obtained from strain gages located at the center of a specimen.

MESSRS. DIETZ, GAILUS, AND YURENKA.—To a considerable extent the comment that the test plotting seems to prove that $x = x$ is correct. However, another set of measurements which it did not seem necessary to include in the paper itself was made.

In that set a Huggenberger extensometer was placed at the central portion of the compression test specimen so as to measure strains over that part of the specimen where the compression is relatively uniform and largely unaffected by the end restraint which occurs where the specimen is in contact with the bearing blocks. Strain measurements made on the Huggenberger resulted in curves completely similar to those reported in the paper although the curves were somewhat displaced (about 14 per cent). It seems, therefore, that the general premise set forth in the paper was substantially correct although the exact shape of the curves might be somewhat modified.

---

[3] C. W. MacGregor, "The Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 508 (1940).

[4] Headquarters, Air Materiel Command, Wright-Patterson Air Force Base, Dayton, Ohio.

METHODS AND EQUIPMENT FOR CONTROLLING SPEED OF TESTING*

By Lawrence K. Hyde[1]

Synopsis

The methods and equipment now in use for controlling speed of testing as represented by rate of application of load or rate of travel of crosshead are discussed briefly. The limitations of these methods as regards reproducibility and relation to rate of straining in the specimen suggest the need for developing more significant methods. A device for controlling rate of straining in the specimen is described which enables the testing machine operator to control the rate of application of load or the rate of travel of the crosshead so as to obtain a predetermined uniform rate of straining throughout the elastic range and as far as desired into the plastic range of the test specimen. There is also described a simple time-interval marking device which records on an autographic stress-strain record the elapsed time in increments of one-tenth of a minute, thus providing a record of the testing speed employed.

About two years ago the Section on Effect of Speed of Testing, under Committee E-1, started to compile information on the capabilities and limitations of then-existing testing equipment with respect to control of speed of testing. In 1947, a report was submitted which includes data by several manufacturers of testing machines on their methods for controlling speed of testing. The purpose of this paper is to review some of these methods with particular reference to some of their capabilities and limitations, and to describe recently-developed equipment that is designed to facilitate further the control and reproducibility of testing speed.

The two principal testing speed control methods employed at the present time are control of rate of application of load and control of crosshead speed (or rate of crosshead separation, or ram travel).

Control of Loading Speed:

Most modern testing machines are equipped with some sort of pacing device by means of which the operator can maintain a predetermined uniform rate of application of load on the test specimen. Such pacing devices take the form of a disk, hand, or pointer, which can be driven at a rate corresponding to the desired uniform rate of application of load, and with which the operator can synchronize the load-indicating hand of the testing machine, thereby obtaining the desired rate of application of load on the specimen. This simple method of testing speed control has the advantage of being independent of the type of testing machine—whether hydraulic or screw driven or other types—or nature of the test set-up with which it is employed, that is, whether the specimen is tested in tension, compression, or flexure. A high degree of reproducibility (up to the proportional limit) of tests so conducted is thus assured. This method of testing speed control has the disadvantage that whereas the relationship between rate of loading and rate of straining is direct until the proportional limit is passed, beyond this point the rate of straining may increase with great rapidity and may attain fantastic values. Furthermore, in the cases of many materials, it is not practicable or even possible to maintain a uniform rate of application of load much beyond the proportional limit, so that testing speed may be uncontrolled during an important phase of the test, such, for instance, as the point on the stress-strain curve used in the determination of yield strength.

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] General Manager, O. S. Peters Co., Washington, D. C.

Control of Crosshead Speed:

Methods for controlling crosshead speed may vary considerably depending upon the type of testing machine. In the case of those in which load is applied by hydraulic means, rate of crosshead (or ram) speed can be adjusted by a graduated valve, whose position indicates the nominal crosshead speed, which may vary under varying conditions of loading. In the case of modern screw power machines, rate of crosshead speed can be adjusted by a graduated dial controlling a variable speed motor. To eliminate the uncertainty of relying on the position of a manual control device as an index of crosshead speed, pacing devices may be used to give the testing machine operator a visual indication of the relationship existing between a predetermined uniform rate of crosshead speed and the actual rate at which the test is proceeding, thereby enabling him to take necessary corrective steps to maintain the actual rate synchronous with the desired rate. Such pacing devices may take the form of a disk, hand, or pointer, which can be driven at a rate corresponding to the desired uniform rate of crosshead speed, and with which the testing machine operator can synchronize an indicating device which shows actual rate of crosshead speed, thereby obtaining the desired crosshead speed. This method of testing speed control has the advantage of being reasonably easily attainable throughout the elastic and plastic deformation ranges of the test specimen, whereas the method of controlling rate of application of load is often attainable only through the range up to the proportional limit of the test specimen. A number of disadvantages are encountered in controlling speed of testing by controlling crosshead speed: rate of straining in the specimen, especially in the tension test, may vary considerably, depending upon the type of test set-up and the type of testing machine; rate of straining in the specimen is not uniform but increases as the load increases, increasing rapidly during the transition from the elastic to the plastic range of the test specimen; since only a small fraction of the crosshead travel appears as strain in the specimen in the tension test, reproducibility of this test as regards straining rate in the specimen, even in the same testing machine, depends on such factors as specimen grips or holders and length of test specimen. Differences in testing machine construction are factors in reproducibility of test conditions as related to straining rate in the specimen when tests are conducted under controlled rates of crosshead speed. Thus in the case of a testing machine in which the position of the sensitive, or weighing, crosshead is fixed relative to the frame of the testing machine, the speed of the moving crosshead relative to the testing machine frame is also a measure of the rate of separation of the two crossheads, whereas in a testing machine in which the sensitive crosshead has appreciable movement relative to the frame of the testing machine, the speed of the moving crosshead relative to the frame of the testing machine is not an accurate measure of the rate of separation of the two crossheads, and this factor can have an adverse effect on reproducibility of conditions involving tests under controlled rates of crosshead speed made on different types of testing machines.

Control of Straining Speed:

By means of a recently-developed device, controlled testing speed in terms of strain in the specimen can be obtained. This strain pacer is particularly well suited to tension testing, where there is no direct relationship between crosshead travel and strain in the specimen, and it is equally adaptable to transverse and compression testing. With its use, uniform straining rate can be maintained through the elastic and as far as desired into the plastic deformation range of the test specimen. The device is adaptable to almost any modern testing machine, and its full utilization requires the use of autographic stress-strain recorder equipment.

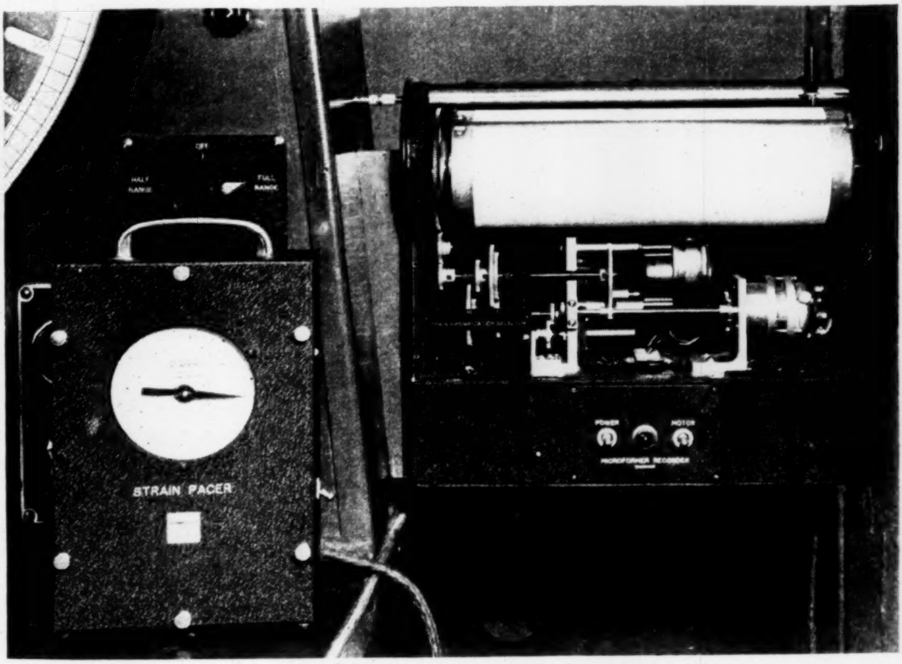


FIG. 1—Stress-Strain Recorder.

The principle of operation and use of the strain pacer is generally similar to that of certain load and crosshead pacing devices already in use. The strain pacer makes use of a hand which can be rotated automatically in proportion to value of strain in the specimen. This hand is mounted coaxially with a dotted disk which can be automatically rotated at a preselected uniform rate. To maintain a uniform straining rate, the testing machine operator controls the speed of the testing machine so that rotation of the hand coincides with that of the disk.

One type of application of the strain pacer is illustrated in Fig. 1, which shows a stress-strain recorder with covers removed to expose the transmitter unit of the strain pacer. The generator unit of a self-synchronous motor-generator com-

TABLE I.—STRAIN PACING SPEEDS.

Strain Follower Type	Strain Magnification	Strain Pacing Speeds, in. per inch per min.					
		0.5 rpm.	0.75 rpm.	1.0 rpm.	1.5 rpm.	2.0 rpm.	2.5 r pm.
A	5	0.05	0.075	0.10	0.15	0.20	0.25
	10	0.025	0.0375	0.05	0.075	0.10	0.125
	20	0.0125	0.00875	0.025	0.0375	0.05	0.0625
B	50	0.005	0.0075	0.010	0.015	0.020	0.025
	100	0.0025	0.00375	0.005	0.0075	0.010	0.0125
	200	0.00125	0.001875	0.0025	0.00375	0.005	0.00625
C	250	0.001	0.0015	0.002	0.003	0.004	0.005
	500	0.0005	0.00075	0.001	0.0015	0.002	0.0025
	1000	0.00025	0.000375	0.0005	0.00075	0.001	0.00125

bination is geared to the recording drum of the stress-strain recorder, so that rotation of the generator is proportional to drum rotation and thus proportional to strain in the specimen. By means of a flexible cable, with polarized plug, the recorder is connected to a small cabinet which can be placed at any convenient location near the testing machine controls. The cabinet contains a dotted disk, rotatably mounted coaxially with the motor unit of the self-synchronous motor-generator combination. The dotted disk is rotated by a synchronous motor connected through a variable gear combination, and six speeds of rotation are obtainable: 0.5, 0.75, 1.0, 1.5, 2.0, and 2.5 rpm. A hand attached to the shaft of the self-synchronous motor rotates in proportion to value of strain in the specimen, and the value of one revolution of the hand, in terms of strain in the specimen, is a combined function of

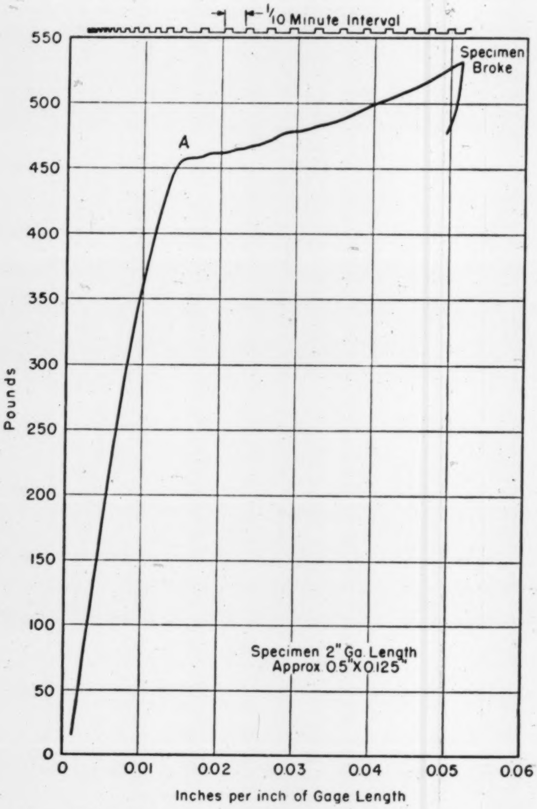


FIG. 2.—Tension Test of Cotton Fabric Polystyrene, Crosshead Speed 0.1 in. per min.

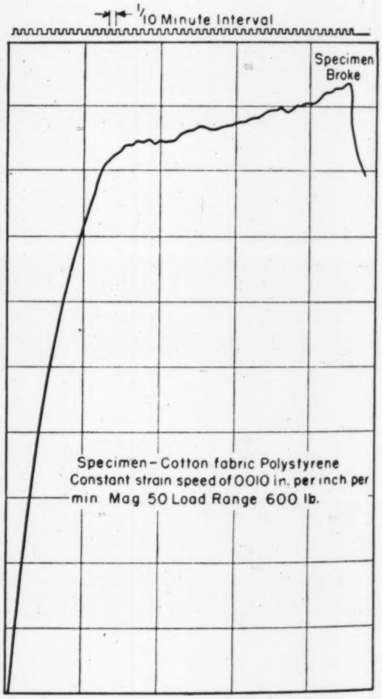


FIG. 3.—Tension Test of Cotton fabric Polystyrene.

the strain follower and of the strain-magnification setting of the recorder. For example, in the case of a 2-in. gage length tension test specimen, one revolution of the hand can represent strain values of from 0.0005 to 0.100 in., which in combination with the obtainable disk speeds can cover straining rates as low as 0.00025 in. and as high as 0.25 in. per min. Table I includes strain-pacing speeds that are obtainable using the pacer in combination with 2-in. gage length strain followers. Other pacing speeds are obtainable at other gage lengths and strain magnifications.

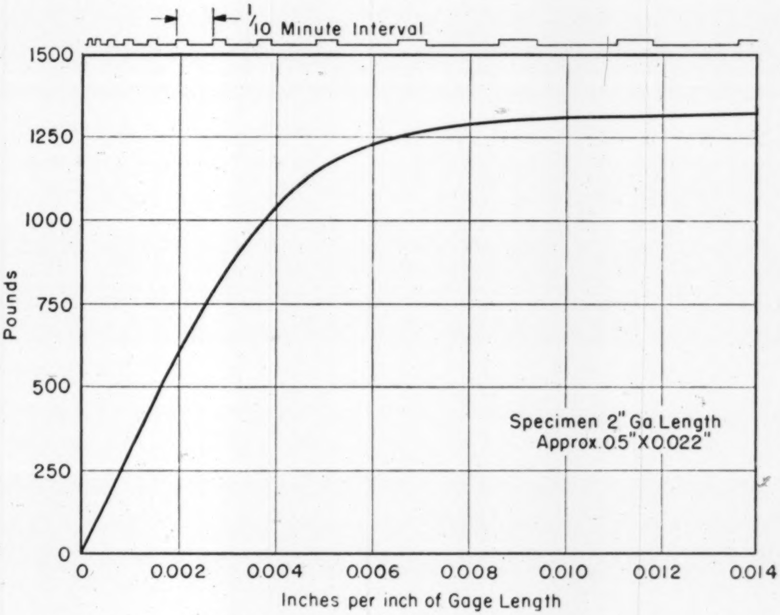


FIG. 4.—Tension Test of Stainless Steel Cross-head Speed 0.100 in. per min.

Time-Interval Marker:

Another recently-developed accessory for use in recording speed of testing is a time-interval marking device which records elapsed time on the autographic stress-strain chart. This device is adaptable to many of the modern stress-strain recorders now in use and records elapsed time in fractional minute intervals, simultaneously with the recording of the stress-strain curve, thus providing a record of the testing speed employed in terms of units of load (or strain) per unit of time.

The time-interval marker consists of a stylus mounted so that it records on the chart on the same time axis as that of the stress-strain recording stylus. A small electro-magnet, mounted near the time-interval marker stylus, and energized at periodic intervals by a synchronously-operated switch, produces a small deflection in the trace made by the stylus.

Autographic Records:

Figures 2 to 8 are stress-strain records illustrating certain features of the strain pacer and the time-interval marker.

Figure 2 is a record of a cotton-fabric polystyrene tested in tension at a uniform crosshead speed of 0.100 in. per minute. The standard 2-in. gage length test specimen was held in Templin grips in a hy-

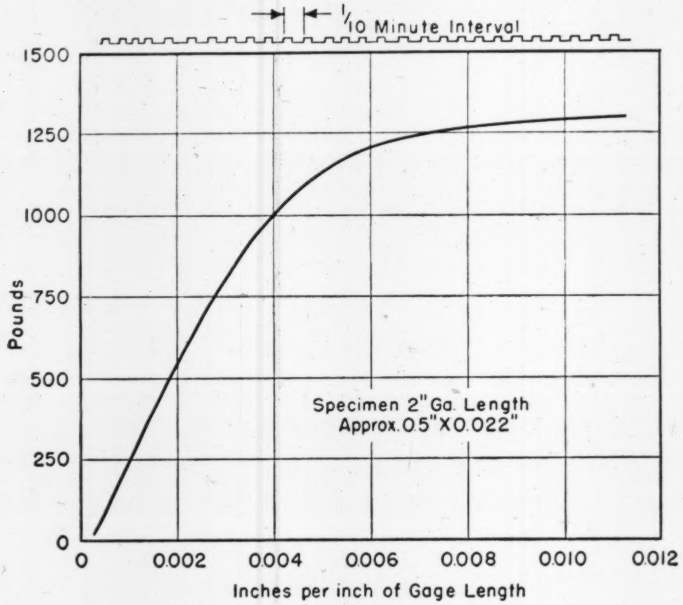


FIG. 5.—Tension Test of Stainless Steel Straining Rate 0.004 in. per min.

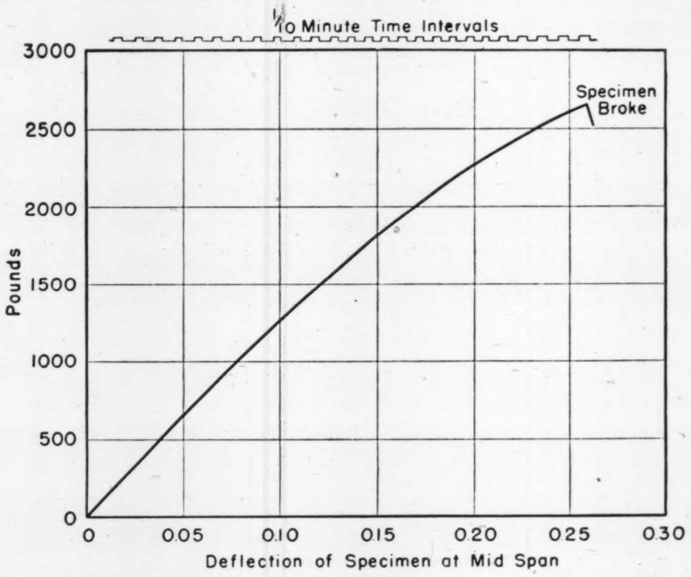


FIG. 6.—Transverse Test on Case Iron Specimen, Testing Speed: 0.1 in. per min. travel of testing machine ram. Transverse test bar 1.2 in. in diameter tested at 18 in. span.

draulic type testing machine in which constant crosshead speed was maintained using one of the pacing devices previously mentioned. It will be observed from the time record that the testing speed, in terms of straining rate, increases up to A, and then levels off at about 0.03 in. (per inch of gage length) per minute, or about 30 per cent of the crosshead speed.

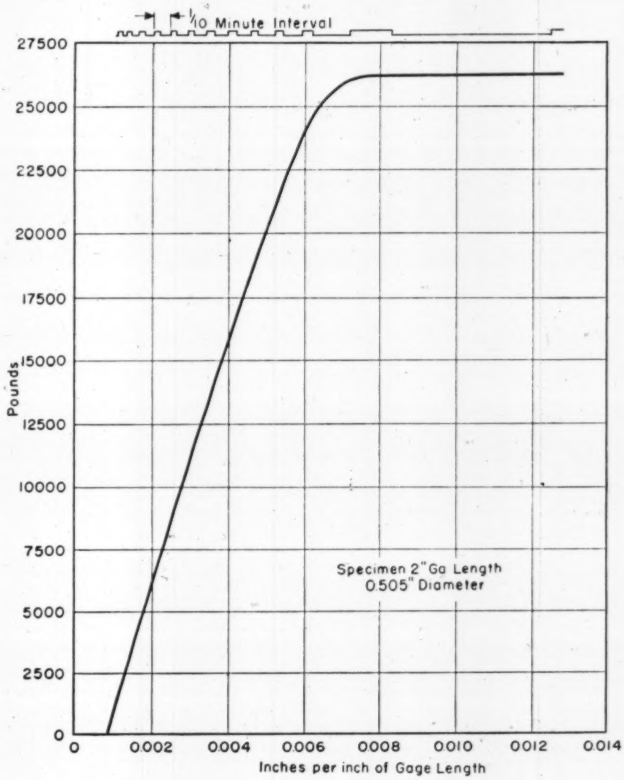


FIG. 7.—Tension **Test on Alloy Steel (Cold Worked) Crosshead Speed 0.10 in. per min.**

Figure 3 is a record of a similar specimen tested in tension at a uniform straining rate of 0.010 in. (per inch of gage length) per minute. The time record indicates that the testing speed in terms of straining rate was substantially uniform throughout the test.

Figure 4 is a record of a stainless steel specimen tested in tension. The specimen was held in Templin grips in a hydraulic type testing machine. During the test a uniform rate of crosshead travel of 0.100 in. per minute was maintained. From the time record it will be observed that the straining rate was increasing continuously throughout the test, and at the end of the test had attained a value approximately 20 times as great as that which obtained at the start of the test. It will also be observed by comparison of Figs. 2 and 4 that although both tests involved 2-in. gage length specimens held in Templin grips, and with all other factors of the test (except specimen material) presumably identical, the straining rates differed greatly.

Figure 5 is a record of a similar stainless steel specimen tested in tension at a uniform straining rate of 0.004 in. (per inch of gage length) per minute. The time record indicates that the testing speed, in terms of straining rate, was substantially uniform throughout the test.

Figure 6 is a record of a transverse test on a specimen of cast iron. The crosshead speed was maintained at a uniform rate of 0.100 in. per minute. The time record indicates that while the testing speed in terms of specimen deformation was not quite uniform, it is for all practical purposes the same as the crosshead speed.

Figure 7 is a record of a cold-worked alloy steel specimen tested in tension at a uniform crosshead speed of 0.100 in. per minute. This was a standard 2-in. gage length, threaded end test bar, tested in a hydraulic testing machine. The time record indicates that the straining rate increased continuously throughout the test and was probably 20 times greater at the end of the test than at the start.

Figure 8 is a record of a standard 2-in. gage length threaded end test bar of brass tested in tension at a straining rate of 0.002 in. (per inch of gage length) per minute. The time record testifies to the uniformity of the straining rate.

Figures 2 to 8 typify a few of the many testing speed problems to which the strain pacer and time-interval marker are adaptable. From Figs. 2, 4, and 7, may be observed some of the variations in straining rate that may be encountered in generally similar tests made in the same testing machine at the same testing speed, when the latter is specified in terms of crosshead speed. In these records the strain pacer was not used, and the time-interval marker furnishes a permanent record of the testing speed as related to stress and strain in the specimen.

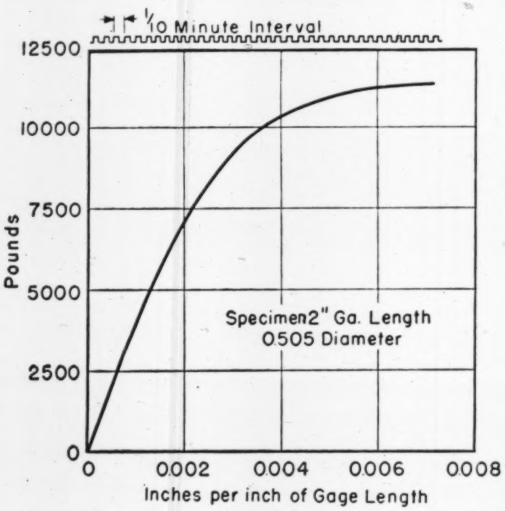


FIG. 8.—Tension Test of Brass Straining Rate 0.002 in. per min.

Figure 6 illustrates one of the few conditions where crosshead travel and measure of specimen deformation are substantially the same. However, in this particular instance it should be noted that in the test covered by Fig. 6, travel of the testing machine ram was substantially the same as specimen deformation measured at midspan, since in the testing machine used in this test the sensitive, or weighing, crosshead had no appreciable movement, whereas in the case of a testing machine in which the sensitive crosshead has appreciable movement, the same crosshead or ram travel would not produce the same rate of specimen deformation.

Figures 3, 5, and 8 illustrate the particular adaptability of the strain pacer for maintaining uniform straining rate in tension tests where considerable disparity exists between crosshead travel and strain in the specimen.

CONCLUSION

From the preceding data it may be seen that the two methods employed at present of controlling testing speed (control of rate of application of load and control of rate of crosshead speed) may be supplemented by a third method (control of rate of straining) which has now reached the stage of practicability. This third method appears to offer interesting possibilities in the direction of developing testing procedures in which conditions in the test specimen are readily reproducible and in which testing speed relates directly to the specimen rather than to some function of the machine in which it is tested.

The new equipment makes it possible to study the effect of rate of straining (within limitations imposed by capabilities of autographic stress-strain recording) on properties of material, and also permits control of testing speed in routine tests of materials which are found to show significant effects, while the time-interval marker provides a permanent record of the speed at which each test was conducted.

DISCUSSION

Mr. F. M. Howell[1] (*presented in written form*).—As the author of this interesting paper points out, most specification requirements concerned with limitations on speed of testing, particularly as applied to the tension test, limit either the rate of stressing or the rate of travel of the crosshead of the testing machine. This is not because these methods are considered the best but primarily because no ready means of controlling the rate of strain has been available.

It is quite generally agreed that the most desirable way of limiting testing speeds in tests of metals is by limiting the rate of strain, that is, by controlling the rate of extension of the specimen within the gage length. The promise of a device that will enable the testing machine operator to control the rate of strain should be of great interest to the testing engineer. The author is to be complimented on the progress that has been made to provide such a device.

---

[1] Chief, Mechanical Testing Div., Research Laboratories, Aluminum Company of America, New Kensington, Pa

THE DETECTION OF CORROSIVE SULFUR COMPOUNDS IN MINERAL TRANSFORMER OIL*

By Frank M. Clark[1] and Edward L. Raab[1]

From the days of its earliest application, the electrical engineer has considered the formation of sludge and the presence of water and sulfur to be the most difficult hazards to the successful operation of the mineral oil-filled transformer. The presence of water has constituted a dielectric hazard. Water is invariably associated with low dielectric strength and ultimate dielectric failure. Its elimination or at least a reduction of its hazard has been the result of the development of the filter press and the centrifuge and careful attention to the proper gasketing of bolted-on covers and plates.

Sludge formation due to oxidation has presented a more difficult problem. But as a result of the combined efforts of the oil technologist and the transformer engineer, the troubles from this source have been substantially eliminated. The use of oxidation inhibitors may offer a further step along the path toward the elimination of this defect in mineral oil.

The problem of sulfur corrosion, however, has received much less attention. The unfortunate results attributable to the use of corrosive sulfur-bearing oils in the early days of the mineral oil-filled transformer clearly defined the danger of free sulfur in the oil. The proper selection of base oils and the development of improved refining techniques have so completely removed free sulfur and the more unstable sulfur compounds that the problem has generally been considered permanently solved. Such, however, is not the case. Sulfur compounds are still present in the oil. The present tendency is toward the operation of the transformer at higher temperatures than have been used in the past. This serves once more to focus attention on the presence and possible hazard to be associated with the sulfur compounds in the oil, which under the lower temperature operation of the past have presented little if any corrosive or dielectric danger.

TABLE I.—DETECTION OF FREE SULFUR IN MINERAL OIL.

Sulfur, per cent by weight	Condition of Copper Surface After 6 hr. Heating at 100 C.	Confirmation Test for Sulfur
0.05	heavy black coating	positive
0.01	heavy black coating	positive
0.005	heavy black coating	positive
0.001	black coating	positive
0.0005	tarnished surface	positive
0.0001	filmed surface	positive

Detection of Free Sulfur

The standard A.S.T.M. Methods D 117[2] have long been accepted as suitable for the detection of "free and corrosive" sulfur in mineral transformer oils. In accordance with the test described therein, 100 ml. of oil are heated at 100 C. for at least five hours in contact with 3 sq. in. (total) surface of copper. Blackening of the copper (confirmed qualitatively for sulfide) is evidence of the corrosive nature of the oil. This test has been completely satisfactory for the detection of free sulfur in mineral oil. As little as 0.0001 per cent free sulfur dissolved in an inert solvent can be so detected (Table I).

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Technical Consultant on Insulation and Chemist, respectively, General Electric Co., Pittsfield, Mass.

[2] Standard Methods of Testing Electrical Insulating Oils (D 117 – 43), 1946 Book of A.S.T.M. Standards, Part III-A, p. 214, Part III-B, p. 65; also Tentative Methods of Testing Electrical Insulating Oils (D 117 – 47T), 1947 Supplement to Book of A.S.T.M. Standards, Part III-A, p. 155, Part III-B, p. 79.

For lower concentrations of sulfur, the copper surface was slightly filmed but was not easily distinguishable from the surface of a similar copper strip heated in like manner in a sulfur-free oil. The use of nitrogen gas to eliminate surface oxidation of the copper during the test might well serve further to increase the sensitivity of the testing procedure.

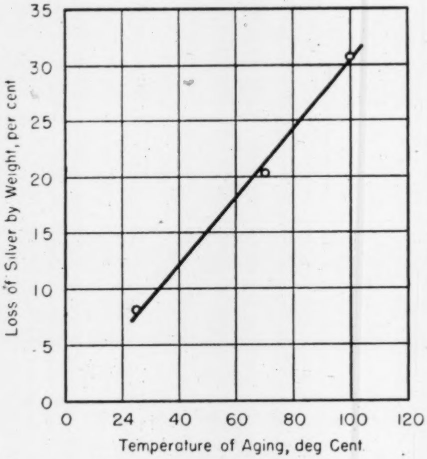


FIG. 1.—The Effect of Temperature on the Corrosion of Silver by Free Sulfur.

Effect of Temperature on Corrosion by Free Sulfur:

To a mineral transformer oil of accepted quality which gave no evidence of sulfur corrosion when tested in accordance with the standard A.S.T.M. method was added 0.20 per cent free sulfur. One hundred milliliters of this treated oil were aged at selected temperatures from 25 to 100 C. with a strip of pure, cleaned silver foil (1 by $1\frac{1}{2}$ in. by 0.005 in.) immersed therein. The effect of temperature on the corrosive action of the sulfur is illustrated in Fig. 1.

Effect of Time on the Corrosion by Free Sulfur:

Although the presence of free sulfur is not easily detectable in mineral oil at concentrations less than 0.0001 per cent when tested in accordance with the A.S.T.M. Tentative Methods D 117-47 T,[2] the action of free sulfur when present in smaller concentrations may produce significant corrosion under the conditions of practical use. In transformer service, the hazard of corrosion is largely determined by the total corrosive sulfur available in the oil in relation to the amount of copper or silver exposed.

In transformers there may be as much as 100 to 150 liters of oil per sq. in. of exposed silver. The total surface of copper exposed to the oil varies widely, but a suitable figure is about 10 sq. in. per liter. Much of this copper surface is embedded in insulation, however, and its effectiveness for sulfur reaction is questionable. The copper surface exposed to the more active attack by sulfur is accepted as not more than 1 sq. in. per liter of oil. In the standard A.S.T.M. test, the conditions are abnormal. In this test a 3-sq. in. surface of copper is exposed to 100 ml. of oil giving a ratio of 30 sq. in. of metal surface per liter of oil. In accordance with the previous paragraph, as little as 0.0001 per cent free sulfur may be detected. This corresponds to 0.0000299 g. of sulfur per square inch of copper.

The sensitivity of detection has been found to be related to the amount of copper or silver exposed per gram of corrosive sulfur present in the volume of oil in which the metal is immersed.

Thus although under the conditions of the A.S.T.M. D 117 test as little as 0.00005 per cent free sulfur is beyond the limit of detection (0.0000149 g. sulfur per square inch of copper), if the amount of the oil used is doubled, thereby increasing the total volume of free sulfur available for reaction, the corrosion produced is detectable.

In like manner, the length of time of the corrosion test is not without effect. Although smaller than 0.0000299 g. of sulfur per square inch of copper is not detectable in the A.S.T.M. test after 5 hr. of heating, there slowly accumulates a tarnish layer on longer heating. First the edges of the copper show attack, then the whole surface becomes filmed with the result that after about 168 hr. of heating at 100 C. the copper surface is covered with a dark deposit which on qualitative analysis gives test for sulfide.

Facts like these call for a more complete description of the sulfur test and raise the question as to its proper duration.

Behavior of this type raises questions concerning the suitability of the present corrosive sulfur test especially with respect to the ratio which exists between the volume of oil used and the copper surface exposed. The requirement of a minimum of 5 hr. heating at 100 C. may well be questioned.

THE TRANSFORMER PROBLEM

The problem of the modern oil filled transformer is not the problem of "free" sulfur. It is the problem presented by the presence of compounds of sulfur which are considered "inert" under the conditions of the present A.S.T.M. test but which become corrosive as a function of the temperature and duration of their commercial use. For the detection of such "potentially" corrosive compounds, the present A.S.T.M. test in Method D 117, is inadequate and misleading.

A well-refined mineral transformer oil of suitable dielectric properties and oxidation resistance contains no free sulfur but does contain up to 0.1 per cent "fixed" sulfur compounds when examined in accordance with the A.S.T.M. Method D 129 - 44.[3] It is with the instability of these "fixed" sulfur compounds that the present problems of sulfur corrosion are concerned. The corrosion normally relates to the susceptibility of copper, but in some instances electrically contacting surfaces are of silver or silver-plated copper. Corrosion "scaling" presents problems for the continued satisfactory operation of the apparatus.

THE CORROSIVE ACTION OF MINERAL OILS CONTAINING "FIXED" SULFUR COMPOUNDS

The metallic corrosion traceable to the action of mineral oils containing sulfur in chemical combination develops as the result of the temperature and duration of contact and varies with the type of sulfur compound present. One type of transformer oil having properties acceptable for commercial use on the basis of the usual dielectric and chemical tests including that for corrosive sulfur (A.S.T.M. Method D 117) illustrates the type of corrosion attributed to the combined sulfur compounds present. Such an oil possessed the following properties:

Color	¼	(D 155 - 45 T)[a]
Neutralization number	0.01 mg. KOH per g. of oil	(D 663 - 46 T)[a]
Saybolt viscosity (37.8 C.)	56 sec.	(D 88 - 44)[a]
Specific gravity (15.5/15.5 C.)	0.856	(D 287 - 39)[a]
Total sulfur (combined)	0.07	(D 129 - 44)[a]
Free sulfur	None	(D 117)[a]

[a] 1946 Book of A.S.T.M. Standards, Part III-A.

This oil when aged in contact with silver or copper at 75 C.—a temperature within the approved operating range of transformer practice—gave evidence of serious sulfur attack. The behavior of this "corrosive" oil is indicated in the following tabulation:

---

[3] Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (D 129 - 44), 1946 Book of A.S.T.M. Standards, Part III-A, p. 284.

Area of metal present	1 sq. in.
Condition of aging	75 C.
Aging container used	sealed glass flask
Volume of oil used	1 liter

Days of Aging at 75 C.	Condition of Metal Surface	
	Copper	Silver
3	unchanged	unchanged
18	slight tarnish film	slight tarnish film
101	heavy tarnish film	heavy tarnish film
125	heavy copper sulfide deposit	flaky silver sulfide formed

It is for the detection and elimination of mineral transformer oils of this type that a suitable test method for corrosive sulfur compounds must be devised. The present test in Methods D 117 is entirely inadequate.

Comparative Corrosion Effect on Silver and Copper:

In the standard A.S.T.M. Test D 117, copper has been used to evaluate the corrosive effect of free sulfur. In many electrical applications silver metal is present. As indicated in the foregoing tabulation, there appears to be but little difference in the detection or the amount of corrosion formed on silver or on copper by the unstable combined sulfur compounds present in the oil. This is substantiated in the following tabulation based on tests conducted at 140 C. in the presence of nitrogen gas, using the same oil as in the previous test:

Metal surface present	1 sq. in. per liter of oil
Volume of oil used	250 ml.
Test container	7 by ½- in. pyrex test tube
Temperature of test	140 C.
Period of heating	48 hr.
Condition of copper after test	Black flakes of copper sulfide
Condition of silver after test	Black deposit substantially similar to the copper test

The corrosion-formed products on the surface of the metals were identified as the sulfide of the copper and silver respectively.

Effect of Temperature:

The corrosive effects produced by fixed sulfur compounds present in mineral transformer oil are increased as the aging temperature is increased from 75 C. to 140 C. Using an oil of the type already described, the effect of temperature is illustrated in the following tabulation and in Fig. 2.

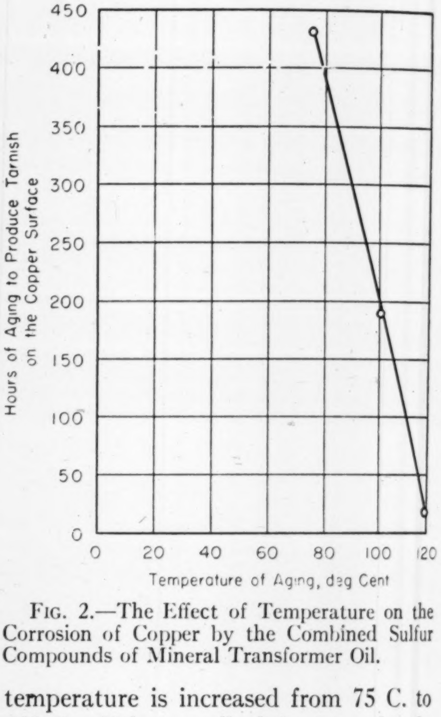


FIG. 2.—The Effect of Temperature on the Corrosion of Copper by the Combined Sulfur Compounds of Mineral Transformer Oil.

Volume of oil	125 ml.
Metal used	Copper
Metal area	1 sq. in. per liter of o
Gas used	Nitrogen
Test container	250 ml. Erlenmeyer flask
Period of heating	as described

Temperature of Aging, deg. Cent.	Tarnish Film Appears on Copper, hr.
75	432
100	192
120	19
140	5

In each test, the film produced on the copper was identified as copper sulfide.

Effect of Surface Area:

With an oil characterized by the severe corrosiveness of its sulfur compounds, the effect of increased metal exposed to the oil during the aging test does not appear to diminish the sensistivity of the test. With oils of a weakly corrosive tendency, the increase in the metal area does tend to diminish the sensitivity of the test. An area of about 1 or 2 sq. in. of metal surface per liter of oil appears best suited for detection purposes. The following tests were carried out in sealed 125 ml. glass flasks, filled to capacity with the oil tested and bubbled with nitrogen gas to remove all oxygen before test. The aging temperature was 140 C. for 19 hr.

Area of Copper Surface Exposed per liter of Oil, sq. in.	Condition of Copper Surface after 19 hr. at 140 C.
TESTS WITH STRONGLY CORROSIVE OILS	
1	Heavy black sulfide flakes
2	Heavy black sulfide flakes
4	Heavy black sulfide flakes
TESTS WITH WEAKLY CORROSIVE OIL	
1	Faint sulfide film
2	Very faint sulfide film
4	Questionable sulfide film[a]

[a] This type of film would not be considered suitable for visual test purposes.

Effect of Oxygen:

The corrosive effects of unstable sulfur compounds present in the oil are reduced when the aging test is carried out under strongly oxidizing conditions. This is illustrated in aging tests carried out in accordance with a procedure similar to that utilized in A.S.T.M. Method D 670 - 42 T[4] for the determination of sludge formation by the high-pressure test. In this test a pyrex test tube (7 by $\frac{1}{2}$ in.) containing 125 ml. oil (similar to that previously used) was heated at 140 C. under 250 psi. gas pressure for 48 hr. in contact with copper (1 sq. in. area per liter of oil). When oxygen was used, sludge was formed on the metal surface. Before checking for sulfur corrosion, this slugde was removed by solvent wash. The results obtained were as follows:

Metal Used	Condition of Metal Surface	
	With Nitrogen	With Oxygen
Copper....	heavy black flakes[a]	faint tarnish film[a]

[a] The film and flakes were identified as copper sulfide.

The effect of oxidation on the sulfur corrosive properties of the oil suggests that an oxidized oil would be characterized by reduced corrosive characteristics. This has been found to be true. The effect may be illustrated with reference to mineral transformer oil after oxidation in accordance with A.S.T.M. high pressure test of Method D 670 - 42 T. For this purpose, the corrosive oil already described was oxidized for 24 hr. at 140 C. under 250 psi. oxygen pressure in accordance with A.S.T.M. Method D 670 - 42 T. The oil after this oxidation was sludged. The sludge was removed and the corrosive characteristics of the oil compared to those of the original oil.

The sulfur corrosion test was obtained by aging for 19 hr. at 140 C. in contact with 1 sq. in. of copper surface per liter of oil. The oil (70 ml.) was placed in a 125-ml. glass flask; the air was removed by nitrogen gas displacement and the flask was sealed. The copper surface of the test demonstrated the reduced corrosiveness of the oxidized oil:

Original Oil.—Copper surface covered with heavy flakes of copper sulfide after test.

Oxidized Oil.—The copper surface was coated with a slight film identified as copper sulfide after test.

---

[4] Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil (D 670 - 44 T), 1946 Book of A.S.-T.M. Standards, Part III-A, p. 946, Part III-B, p. 628.

Effect of Inhibitors:

The effect of oxidation inhibitors on the corrosivity of the oil is not clear. In no case did the presence of an inhibitor increase the corrosivity of the oil, and in some instances a distinct decrease in the corrosivity was noted. In no instance, however, was the corrosivity entirely eliminated.

Effect of High Conductor Temperature:

A conductor carrying heavy current immersed in a mineral oil containing unstable "fixed" sulfur compounds may be seriously corroded despite the fact that the oil otherwise meets the usual requirements, including the free sulfur test of A.S.T.M. Method D 117, generally recognized as indicative of good and acceptable oil. This may be demonstrated in a set up using either copper or silver conductor.

Silver foil (1¼ by ⅛ in.) having an exposed area of 0.3125 sq. in. was immersed in 313 ml. of transformer oil contained in a 600-ml. beaker. Thirty amperes of 60-cycle current were passed through the foil which was placed about ½ in. from the bottom of the container. The ratio of silver surface to oil volume was 1 sq. in. per liter. The test was carried out in a covered box to eliminate light effects. At the temperature equilibrium established as the result of the 30 amp. of current passed through the foil, the foil surface temperature was 107 C. The temperature of the body of the oil was established at about 60 C.

Under the conditions of this test, the surface of the silver foil became gradually covered with a deposit which was identified as silver sulfide:

Days of Test	Condition of Silver Foil Surface
3............	black film formation begins
6............	entire foil surface covered by film
8............	heavy black film formed on foil surface
12............	black deposit increased in amount

During the test as described, no significant change was observed in the oil, the color of which had increased to approximately one and one-half (A.S.T.M. Method D 155)[5] and the neutralization value to 0.014 mg. of potassium hydroxide per gram of oil.

Effect of Voltage:

Voltage application has detectable effect on the corrosivity of mineral transformer oils containing combined sulfur compounds. A typical transformer oil containing 0.07 per cent combined sulfur and no detectable amount of free sulfur was submitted to 60-cycle a-c. voltage. This test set-up consisted of silver foil electrodes mechanically spaced to give the dielectric stress indicated in the following tabulation under the 3250 v. a-c. applied during immersion in the oil at 75 C.; 400 ml. of oil were used. The test electrodes were 0.003 in. thick and 2¼ sq. in. in area.

CONDITION OF OIL.

Dielectric Stress Applied, volts per mil	Period of test, days	Color	Neutral Number	Power Factor 25 C., per cent	Condition of Silver Electrodes
0	81	¾	0.018	0.447	slight tan film
13	81	1	0.014	0.407	thin tan film
26	81	1	0.014	0.532	black film
52	81	1¼	0.062	0.900	heavier black film

In all instances, the film observed on the silver electrodes was identified as the metallic sulfide.

Similar tests at 25 C. showed substantially no sulfur corrosion irrespective of the voltage stress applied. The electrical changes in the oil however paralleled those observed in the 75 C. tests described.

---

[5] Tentative Method of Test for Color of Lubricating Oil and Petroleum by Means of A.S.T.M. Union Colorimeter (D 155 – 45 T), 1946 Book of A.S.T.M. Standards, Part III-A, p. 839.

Dielectric Stress Applied, volts per mil	Days of Test	Power Factor of Oil at 25 C., per cent
0	81	0.468
13	81	0.420
26	81	0.56
52	81	0.86

A SUGGESTED TEST FOR CORROSIVE COMBINED SULFUR

A suitable procedure for detecting the presence of corrosive (combined) sulfur compounds in mineral oil consists in heating the oil in the presence of a clean copper surface for 19 hr. at 140 C. The corrosion of the copper surface at the end of the test is accepted as a measure of the corrosivity of the oil. For a more exact evaluation, the copper sulfide formed may be quantitatively measured and expressed in terms of the copper surface area exposed to the oil.

In this test, a 250-ml. ground glass stoppered Erlenmeyer flask or bottle is used. In 250 ml. of the oil to be tested, is placed a strip of mechanically cleaned copper ($\frac{1}{4}$ by 1 in.) bent to form a *V* shape. The bent copper strip is placed on edge in order to expose the total metal surface to the oil. Dry nitrogen gas is then bubbled through the oil for 1 to 2 min. in order to displace the air in the gas space over the oil. The glass stopper is then quickly inserted in the flask or bottle and the oil and copper are heated for 19 hr. at 140 ± 2 C. After the aging run, the copper strip is removed from the oil and washed with acetone or other suitable solvent to remove the oil. A filmed or tarnished metal surface is indicative of the presence of corrosive sulfur in the oil. The presence of sulfur may be confirmed by the treatment of the metal with hydrochloric acid and the detection of hydrogen sulfide by means of lead acetate paper in the usual manner. To determine whether the sulfur corrosion of the copper is due to free sulfur or to chemically combined sulfur compounds which have heretofore been considered as possessing suitable stability for transformer use, it is necessary to run a test in accordance with the present A.S.T.M. Method D 117 procedure. Free sulfur or "unstable" sulfur compounds are easily detectable with this testing procedure.

Effect of Oil Treatment:

In the application of the testing method suggested for evaluating the corrosivity of mineral oils containing combined sulfur, it has been established that exhaustive treatment of the oil with fullers earth produces no substantial effect on the corrosivity of the oil. For this purpose an oil of substantial corrosive characteristics under the conditions of the suggested test was treated with 3 per cent of its weight of 80/300 mesh fullers earth at 50 to 75 C. After several such treatments the corrosivity of the oil was apparently undiminished. Treatment of the oil with fullers earth by the percolation method gave similar results:

Oil Tested	Condition of Copper Surface After Application of Suggested Testing Procedure
Original................	heavy black flakes formed
Fullers earth treated.....	heavy black flakes formed

Vacuum treatment of the oil at 100 C. under $5\,\mu$ pressure gave no improvement:

Oil Tested	Condition of Copper Surface After Application of Suggested Testing Procedure
Original oil..............	heavy black flakes formed
Vacuum treated oil......	heavy black flakes formed

Caustic wash or treatment with concentrated sulfuric acid produced no substantial change in the corrosivity of the mineral transformer oil:

Oil Tested	Condition of the Copper Surface After Application of the Suggested Testing Procedure
Original oil..............	heavy black flakes formed
Caustic washed..........	heavy black flakes formed
Sulfuric acid washed.....	heavy black flakes formed

Application of the Suggested Test for Corrosive Combined Sulfur:

The test procedure suggested for the detection of corrosive combined sulfur has been applied to a variety of mineral transformer oils, all of which fully meet the requirements of the present A.S.T.M. Test D 117 for free and corrosive sulfur but most of which show corrosive characteristics to some degree in the test proposed. Typical test results follow:

Oil	Specific Gravity at 15.5 per 15.5 C.	Condition of Copper After Test	Qualitative Test for Sulfide in the Film Formed
No. 1....	0.883	slight tarnish	positive
No. 2....	0.854	heavy flakes	strong positive
No. 3....	0.869	heavy flakes	strong positive
No. 4....	0.890	slight tarnish	positive
No. 5....	0.872	slight film	positive
No. 6....	0.889	faint film	slight positive
No. 7....	0.891	faint film	slight positive
No. 8....	0.878	slight film	positive

The visual appearance of the copper surfaces after test is illustrated in Fig. 3.

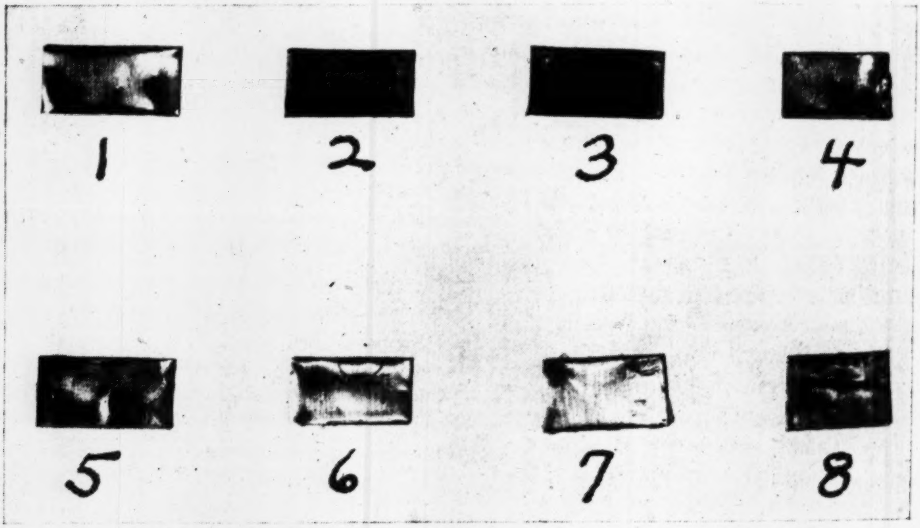


Fig. 3.—Illustration of the Corrosive Characteristics of Eight Different Commercial Mineral Transformer Oils when Tested with Copper in Accordance with the Suggested Procedure.

Since substantially all types of mineral transformer oil contain combined sulfur and give copper corrosion to some degree as indicated in the foregoing tabulation, a suitable test must evaluate the corrosivity of the oil in quantitative terms for which a method of procedure must be established and acceptable limits formulated. One such method has been developed by the Consolidated Edison Co. of New York, Inc. A description of this method is appended through the courtesy of Mr. E. R. Thomas, Test Engineer of the Consolidated Edison Co.

Acknowledgment:

The authors wish to acknowledge the assistance of Mr. S. W. Kernaghan of the Pittsfield Laboratory of the General Electric Co., whose help was greatly appreciated in the study of the broader aspects of the problem of which the data of this paper are a part.

APPENDIX

SPECTROPHOTOMETRIC DETERMINATION OF THE AMOUNT OF SULFUR DEPOSITED ON THE COPPER STRIPS IN THE GENERAL ELECTRIC METHOD OF TEST

Reagents and Apparatus

1. Hydrochloric acid 2:1. Mix 700 ml. of concentrated hydrochloric acid with 350 ml. of distilled water.
2. Neutral zinc acetate solution 0.6 per cent. To 300 ml. of stock solution of two per cent neutral zinc acetate, add 700 ml. of distilled water.
3. Strips of lead acetate paper.
4. Diamine reagent. Dissolve 0.04 g. of para amino dimethyl aniline hydrochloride in 100 ml. of 1:1 hydrochloric acid. (See Note 1.)
5. Ferric chloride solution 0.02 molar. Dilute 20 ml. of 0.1 molar stock solution to 100 ml. with distilled water. To prepare stock solution, dissolve 27 g. of ferric chloride crystals in 500 ml. of concentrated hydrochloric acid and dilute to 1 liter with distilled water.

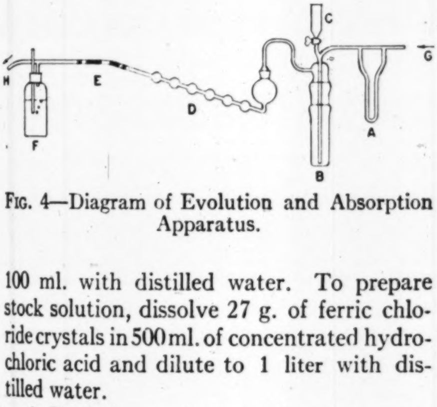


Fig. 4—Diagram of Evolution and Absorption Apparatus.

6. Evolution and absorption apparatus. (See Fig. 4.)
7. Coleman spectrophotometer, type 11.
8. Nitrogen gas.
9. Volumetric flask, 200 ml.
10. Graduated cylinder, 25 ml.
11. Transfer pipette, 10 ml.
12. Transfer pipette, 2 ml.

Procedure

1. Assemble the evolution and absorption apparatus as shown in Fig. 4.
2. Place 25 ml. of 0.6 per cent zinc acetate solution in the Meyer absorption tube, and a strip of lead acetate paper in the check tube. (See Note 2.)
3. Place the sample in the evolution tube and pass nitrogen through the apparatus at a rate of 75 ml. per min. (See Note 3.)
4. Add about 10 ml. of hydrochloric acid 2:1 through the funnel, and allow the action to proceed for 30 min.
5. Wash the contents of the absorption tube into a 200 ml. volumetric flask and dilute to about 180 ml. with distilled water.
6. Add 10 ml. of diamine reagent and mix gently. (See Note 4.)
7. Add 2 ml. of ferric chloride 0.02 molar, and mix thoroughly.
8. Dilute to 200 ml., mix thoroughly, and allow to stand at least 15 min.
9. Transfer a portion of the solution to a 13-ml. cuvette, and determine the per cent transmission on the Coleman Spectrophotometer, using 670-mμ wave length and a PC-5 filter. (See Note 5.)
10. Read parts per million hydrogen sulfide on the curve provided. (See Note 6.)

Calculations

Parts per million hydrogen sulfide × 0.2 = mg. hydrogen sulfide in sample.

Note 1.—The diamine reagent should be freshly prepared and used within 24 hr.

Note 2.—If the lead acetate paper shows a brown or black stain during the test, hydrogen sulfide has not been absorbed completely. The test should be discarded.

Note 3.—The sample will be a more or less discolored copper strip obtained in the General Electric Test for Corrosive Sulfur Compounds in Transformer Oil. The copper strip has been thoroughly rinsed with acetone to remove transformer oil.

Note 4.—The temperature of the solution during the development of the color should be about 25 C.

NOTE 5.—The per cent transmission is determined by the null-balance method. Distilled water may be used as a blank.

NOTE 6.—If the per cent transmission is too low, the determination may be made by diluting the test solution and taking another reading and correcting the calculation accordingly. If it is known before testing that the hydrogen sulfide will be too high, step 5 in the procedure may be changed to transfer the absorbent to a larger flask and using proportionately greater amounts of diamine and ferric chloride reagents.

REFERENCES

(1) Foster D. Snell and Cornelia I. Snell, "Colorimetric Methods of Analysis," D. Van Nostrand and Co., Inc., New York, N. Y., Vol. I, p. 593 (1936).

(2) John H. Yoe, "Photometric Chemical Analysis," John Wiley and Sons, Inc., New York, N. Y., Vol. I, p. 373 (1928).

(3) L. H. Almy, "A Method for the Estimation of Hydrogen Sulfide Proteinaceous Food Products," *Journal*, Am. Chemical Soc. Vol. 47, p. 1381 (1925).

DISCUSSION

Mr. G. H. von Fuchs.[1]—This paper is a great step forward and will be extremely helpful to refiners of transformer oil. I am pleased to see that a distinction is being made between total and corrosive sulfur. I should like to ask the authors a few questions regarding this work: Is there any correlation between total sulfur and corrosive sulfur? I would not expect any relationship at all.

With respect to refining, on which I can speak with some information, I should like to say that absorption on clay is out of the question. There is no use in trying clay for this purpose, at least clay as we know it today.

On vacuum treating the oil, the authors assume that the sulfur compounds with which we are dealing are more volatile than the oil itself, which is not the case. There are some sulfur compounds that have the same volatility as the oil. By distillation we may remove certain reactive sulfur compounds from the low-boiling fractions of gasoline, but beyond that it is not possible to go.

Sulfuric acid is a more potent agent in this case, and if the authors would give the quantity and the concentration or acid strength used, I might be able to comment, because I am rather certain that some real potent sulfuric acid may work, but of course, I am not recommending it.

The coating formed on metallic copper and silver referred to is termed the sulfide. According to my theory on copper bearings in motor cars, I would say that most of this coating is not sulfide but a metal-organic sulfur complex, possibly a mercaptide. There are some patents which refer to the addition of sulfur compounds in the motor oil to cut down corrosion of the bearings. If the protection is properly carried out, even a corrosive oil—in this case I am thinking of acid corrosion—will not attack those bearings for a considerable period of time. Again I am not recommending it; I am just mentioning it.

I think it is an excellent precaution to use a nitrogen blanket and thus prevent oxidation during the corrosion test, because corrosive sulfur compounds, being extremely active, have a tendency to oxidize to noncorrosive forms. The sulfur, which is at first tied onto a hydrocarbon radical, has a great tendency to exchange the carbon bonds for oxygen bonds, and then we have various sulfones, sulfonic acids, etc., which, of course, would not produce a sulfide film.

The effect of oxidation inhibitors is very interesting. A good oxidation inhibitor or anti-oxidant in this case would have no effect whatever on the corrosivity of the oil. It may, however, make the blanket unnecessary because it will prevent the oxidation of the sulfur compounds which would normally occur in the absence of an inhibitor, and which necessitates the use of the nitrogen blanket. I would predict, having made no test in this direction, that if the right kind of inhibitor were used, the nitrogen blanket would not be necessary, which would simplify the procedure quite a bit.

---

[1] Shell Oil Co., Wood River, Ill., at present Consulting Chemical Engineer, Niagara Falls, N. Y.

MR. E. T. SCAFE.[2]—We have observed some loss in weight of copper specimens during corrosion tests with certain petroleum products. I should like to ask if that type of information was available in this test with transformer oils and, if so, were there any relationship between the degree of corrosion and loss in weight of copper test piece.

MR. L. B. SCHOFIELD.[3]—This very timely paper clearly indicates the urgent need for a test which will detect corrosive sulfur compounds in transformer oil. We have no standard test method at present which is adequate for this purpose. Hence I should like to emphasize two points regarding the paper from the utility operating and maintenance viewpoint.

First, our company has a purchase specification for new insulating oil which includes a stipulation that the oil shall show no evidence of corrosive sulfur when tested in accordance with the present A.S.T.M. standard test for so-called "free and corrosive" sulfur.[4] For many years, prior to approximately the last two years, we experienced no failures in this free and corrosive sulfur test.

In recent years, however, the record is not so good. One tank car has been rejected because of corrosive sulfur. Also we have found several marginal cases, where the present A.S.T.M. test would not indicate the corrosivity of certain oils which we had reason to believe should be held in question. In these cases, by moderate exaggeration in the time period of the present A.S.T.M. test at 100 C., we were able to get indications of a corrosive sulfur component in the oil. While these indications were not sufficient to cause rejection of these shipments, they caused us no little anxiety. Certainly the electric utility cannot tolerate an oil with corrosive sulfur, free or combined, and we must have a test we are sure would prevent such an oil getting into service.

Secondly, there has been some question, particularly on the part of the oil producers, as to just which of the consumers are interested in more sensitive and dependable tests for corrosive sulfur. I can only say, as a representative of one utility, that we are very much concerned, chiefly because of the growing trend in the up-rating of transmission equipment, which is presently available to take care of ever-increasing load on the system. This means that proportionately the load on equipment, with respect to past practices, is being substantially increased, causing an increase in operating temperatures or higher oil temperatures. This is one very good reason why a very urgent need exists for developing an improved test for corrosive sulfur in oil, in general accordance with the procedure advocated by the authors.

MR. E. A. SNYDER.[5]—As Chairman of Subcommittee IV of Committee D-9, I want to go on record by stating that our subcommittee was not fast asleep. As soon as we found out several years ago about the trouble that was being experienced in this way, the matter was referred to our Section S on Sulfur, and it has been steadily at work in an endeavor to provide an improved test method which will, we hope, eliminate oils that are at present giving trouble with sulfur.

We also, at the same time, revised the present standard free sulfur test method to an extent which we feel sure would have eliminated most of the oils that gave sulfur trouble. It is revised to an extent that it will reveal not only the presence of so-called free sulfur, but also some of the more easily reactive sulfur compounds that in the course of operation might produce a sulfur corrosion problem.

---

[2] Supervising Analyst, Socony-Vacuum Laboratories., Paulsboro, N. J.

[3] Materials Engineer, Commonwealth Edison Co., Chicago, Ill.

[4] Standard Methods of Testing Electrical Insulating Oils (D 117 - 43), 1946 Book of A.S.T.M. Standards, Part III-A, p. 214; Part III-B, p 65.

[5] Specialist on Electrical Insulating Oils and Marketing Assistant, Chemical Products Div., Lubricating Dept., Socony-Vacuum Oil Co., Inc., New York, N. Y.

I feel that this was a very timely paper, and I want to congratulate the authors on an excellent and instructive presentation of the subject.

MR. F. M. CLARK (*author's closure*).—Like Mr. von Fuchs, we did not expect and did not observe on test any relationship between the total sulfur content of an oil and its corrosive action on copper or silver. Some oils with the lowest total sulfur content were to be classed among the most corrosive of those tested. It is the type of sulfur compound present in the oil, rather than the total amount, which is the important factor in determining the metallic corrosion obtained. These are most easily removed by exhaustive treatment with fuming sulfuric acid. In the tests with sulfuric acid described in the paper, commercially concentrated sulfuric acid was used at room temperature.

We have not attempted to analyze the "sulfide" deposit formed on the metal aside from identifying that the material is a sulfur compound. Mr. von Fuchs suggestion that the deposit is a metal-organic sulfur complex warrants further study. Preliminary observations, however, do not support this suggestion.

We also have observed, confirming Mr. Scafe's results, that corrosion of copper by sulfur containing oils is accompanied by a loss of weight but we have not established a quantitative relationship.

Both Mr. Snyder and Mr. Schofield refer to the present 6-hr. 100 C. test for free and corrosive sulfur. This test has been found to be satisfactory for the detection of free sulfur in the oil. The present-day oil problem, however, rarely concerns the presence of free sulfur. Our experience indicates that the corrosivity due to sulfur in oil is invariably due to combined sulfur, and it is indeed the exception that oil which is considered by practical engineers as unfit for transformer use will show excessive corrosion on the present test. We are inclined to differ with Mr. Snyder in his belief that the present test can give satisfactory protection against the use of hazardous oils. We have attempted to obtain such protection by increasing the ratio of oil volume to metal surface area to a ratio more nearly equal to that of the average transformer practice and by increasing the temperature. Our experience indicates also that the present test is unsatisfactory because of the possibility of oxidation which we, like Mr. von Fuchs, have found to decrease the reliability of the test by the conversion of the corrosive sulfur compounds to sulfones or other non-corrosive oxygen containing sulfur compounds.

It is our hope that the proper committee of the American Society for Testing Materials will study our suggested testing procedure and supplement it with an adequate method for quantitatively determining the total amount of the sulfide corrosion product found.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON FUNCTIONAL TESTS FOR BALL BEARING GREASES

At the Eighth Session of the Fifty-first Annual Meeting of the American Society for Testing Materials held in Detroit, Mich., on June 23, 1948, the Symposium on Functional Tests for Ball Bearing Greases was held under the sponsorship of Technical Committee G on Lubricating Grease of Committee D-2 on Petroleum Products and Lubricants. The soundness of the principle of performance evaluation of greases has been generally accepted, but agreement on procedures for such evaluation had not yet been reached. In an effort to standardize such tests for ball bearing greases, a committee of the Society has found a variety of methods from which to choose. To aid their selection, this symposium was arranged so that users of several methods can describe the degree of their successes. Five papers were prepared and included in the Symposium:

Development of Functional Grease Test Methods for the Aircraft Industry—D. H. Moreton
Grease—An Oil Storehouse for Bearings—D. F. Wilcock and Marshall Anderson
Laboratory Performance Tests for Antifriction Bearing Greases—M. Herbst, W. A. Prendergast, R. S. Barnett, W. J. Finn, and O. P. Puryear
Service Experience with Grease—W. T. Everitt and C. L. Pope
Factors Affecting Simulated Service Tests of Greases—S. M. Collegeman and John R. Belt

These papers together with discussion have been issued as A.S.T.M. Special Technical Publication No. 84 entitled "Symposium on Functional Tests for Ball Bearing Greases."

SUMMARY OF PROCEEDINGS OF THE FORUM ON TRACTOR FUELS

The Forum on Tractor Fuels was sponsored by Technical Committee L on Tractor Fuels of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants and was held in Chicago, Ill., January 9, 1948. The purposes of the Forum were to lay the ground work and explain the field to be considered in the work of Technical Committee L. The following five papers were included:

Tractor Statistics as Related to the Farmer—H. E. Everett
The Farmers' Interest in Tractor Fuels—E. L. Barger
A Review of Tractor Requirements as Related to Fuels—Raymon Bowers
The Complexity of Tractor Fuels and Taxation—Arthur B. Anderson
The Effect of State Tax Regulation and State Specifications on Tractor Fuels from the Petroleum Industry Standpoint—C. N. Hinkle

These papers and the discussion have been issued as a special Technical Publication No. 82, entitled, "Forum on Tractor Fuels."

SUMMARY OF MEETING ON PETROLEUM PRODUCTS AS RELATED TO AUTOMOTIVE EQUIPMENT

At a Joint Meeting of the Society's Detroit District Council and A.S.T.M. Committee D-2 on Petroleum Products and Lubricants, held in Detroit, Mich., in October, 1947, the following two papers were presented:

Petroleum Lubricating Oils as Related to Automotive Equipment—C. M. Larson
The Efficient Production and Utilization of Motor Gasoline—W. M. Holaday

Because of the magnitude of the oil and automotive industries and their importance to the American way of life, it was felt that the Society should encourage cooperation between these two industries and to promote developments that will increase their efficiency. It was with these thoughts in mind that the program for the meeting was developed.

The two papers were made available as a separate pamphlet, A.S.T.M. Technical Publication No. 81, entitled, "Petroleum Products as Related to Automotive Equipment."

THE EFFECT OF TEMPERATURE ON THE CREEP OF TWO LAMINATED PLASTICS AS INTERPRETED BY THE HYPERBOLIC-SINE LAW AND ACTIVATION ENERGY THEORY*

By W. N. Findley,[1] C. H. Adams,[2] and W. J. Worley[1]

Synopsis

Results of creep tests of a canvas laminate and a rayon laminate at temperatures of 42.5, 77, and 128.4 F. are reported. The relative humidity of the air surrounding the specimens was maintained at about 50 per cent by special apparatus described. Specimens were tested at several different stresses for each of the three temperatures. Dimensional changes under no stress and changes in weight of the material were recorded as well as creep.

It was observed that the canvas laminate was inferior in creep resistance to the rayon laminate at all temperatures tested, and the rate of creep of the canvas laminate increased much more rapidly with increasing temperature than the rate of creep of the rayon laminate.

The applicability of the hyperbolic sine law for creep rate *versus* stress and the activation energy theory for creep are demonstrated for the test data obtained from the canvas laminate.

Many creep tests on plastics and plastic molding materials and laminates (**1-8**)[3] have included creep tests at room temperature only. Some of the creep tests of plastics and plastic molding material that have been conducted at different temperatures, but under conditions in which the relative humidity was not controlled, are given in earlier papers (**9-14**). The need for further creep studies of plastics, investigating a range of temperatures and humidities as well as stresses, has been felt for some time. In particular as suggested in the 1944 paper by Findley (**15**) creep studies over a range of temperatures and stresses at constant relative humidity have been needed in order to test the validity of the activation energy theory as applied to the creep of plastics.

The present investigation was conducted in 1945 to determine the creep behavior of two plastic laminates at three different temperatures under conditions of constant load, temperature and relative humidity.

Materials Tested and Specimens

Two laminated plastics were tested in this program: a canvas laminate molded under the relatively low pressure of 180 psi. with a phenol formaldehyde resin; and a rayon laminate of the saponified acetate type molded at a pressure of 1100 psi. with a phenolic resin. The preparation and composition of the laminates tested are summarized in Table I, while some of the mechanical

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Research Associate Professor and Instructor in Theoretical and Applied Mechanics, respectively, College of Engineering, University of Illinois, Urbana, Ill.

[2] Research Laboratory, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1239.

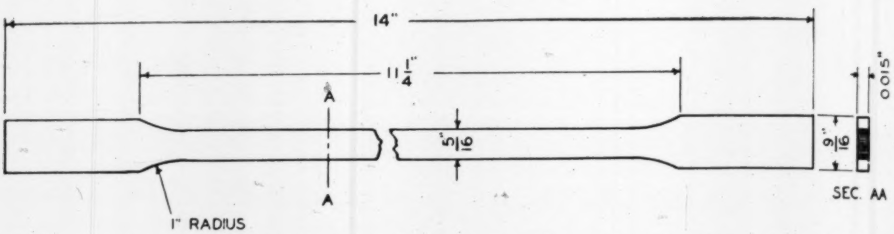


FIG. 1.—Creep Specimen as Machined from the Laminated Sheet (The Cross Hatch Lines in Section AA. Indicate the Direction of the Laminations).

TABLE I.—PREPARATION AND COMPOSITION OF THE LAMINATES.

Designation	Low-pressure-molded Canvas Laminate	Rayon Laminate
Laminator	Synthane Corporation	Formica Insulation Company
Manufacturer of resin	Bakelite Corporation	Ironsides Company
Number of resin	BV-16887	91-L
Type of resin	Phenol-formaldehyde	Phenolic
Fabric	Army duck	Celanese WE-3975
Warp	Cotton	Special high tenacity Fortisan (Saponified acetate)
Woof	Cotton	Cotton
Weave	Square	3/1 Twill
Weight raw of fabric	10.38 oz. per sq. yd.	12.5 oz. per sq. yd.
Thread count of fabric	38 by 52	12 by 75
Tensile strength of warp	160 lb. per in.	1100 lb. per in.
Tensile strength of woof	120 lb. per in.	
Elongation at break		10% at relative humidity of 63% and temperature of 74 F.
Number of plies in laminate	32	23
Per cent resin by weight in body of laminate	51	37 to 40
Lay of laminations	Cross laminated	Cross laminated
Molding temperature	320 F.	320 F.
Molding pressure	180 psi.	1100 psi.

TABLE II.—A SUMMARY OF THE MECHANICAL PROPERTIES OF THE LAMINATES AS GIVEN IN A PREVIOUS REPORT (**8**).

Material	Low-Pressure-Molded Canvas Laminate	Rayon Laminate
Specific gravity	1.29[a]	1.37[a]
Tension modulus, psi. × 10^{-6}	0.81	1.78
Ultimate strength in tension, psi.	8 300	25 600
Tension yield strength at 0.05 per cent offset, psi.	3 800	7 700
Tension yield strength at 0.2 per cent offset, psi.	5 100	10 800
Compression modulus, psi. × 10^{-6}	0.94	1.68
Ultimate strength in compression, psi.	18 300	20 100
Compression yield strength at 0.05 per cent offset, psi.	3 200	7 200
Compression yield strength at 0.2 per cent offset, psi.	5 100	9 600
Rockwell hardness perpendicular to laminations	M 95	M 98
Fatigue Strength at 77 F.[b] psi.	2 700	5 800

[a] Data taken from tests made at the National Bureau of Standards.

[b] Fatigue strength at 10^7 cycles for unnotched specimens tested in a rotating cantilever beam fatigue machine.

properties of the two laminates are summarized in Table II. A more complete description of the preparation and composition of the laminates and of their mechanical properties is given in a paper by Findley and Worley (**8**). Specimens for the present investigation were taken from the same sheet of laminate used in collecting the data reported in the above reference.

The shape of specimen used is shown in Fig. 1. A milling cutter was used to cut the creep specimen from the original sheet in such a manner that the flat side of the specimen was perpendicular to the plane of the sheet. The reduced section and the radii at the end were formed on a shaper. The direction of the longitudinal axis of the creep specimen was parallel to the axis of the specimens used in the previously reported tests of these materials and was parallel to one of the axes of the fabric from which the cloth laminated piece was prepared.

The machined surfaces of the specimens were finished with No. 3 emery cloth and were placed in an air-conditioned room maintained at 77 F. and 50 per cent relative humidity. All specimens were allowed to condition for several months in this room prior to testing. Control specimens used to determine the magnitude of shrinkage during the test were identical with the creep specimens. The control specimens for determining the change in weight of the material during the test were the same as the creep specimens except that the width throughout corresponded to that of the reduced section of the creep specimens.

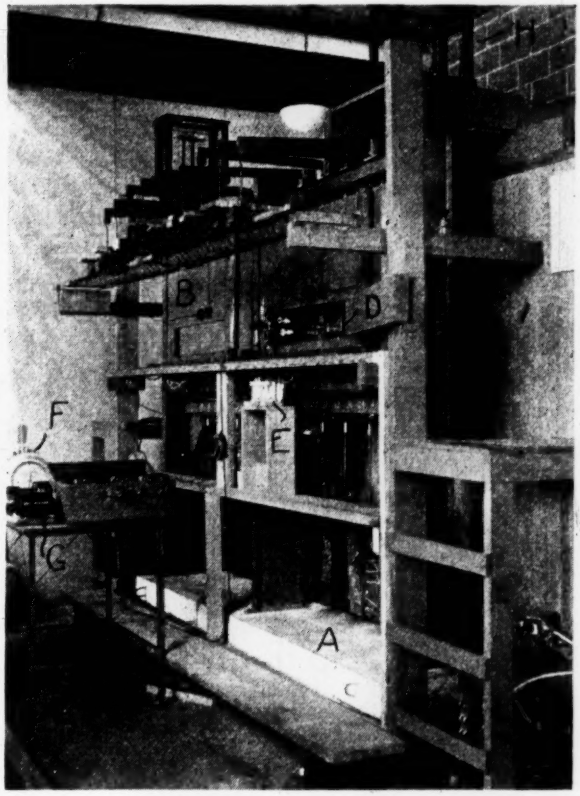


FIG. 2.—Apparatus for High and Low Temperature Creep Tests.

TEST APPARATUS—TEMPERATURE AND HUMIDITY CONTROL AND MEASUREMENT

The creep testing equipment consisted of two steel racks on which all specimens were tested simultaneously. One of these racks was placed in the air-conditioned room, previously mentioned, which was maintained at a temperature of 77 ± 1 F. and a relative humidity of 50 ± 2 per cent. The other rack was located in a room without air conditioning. Two insulated cabinets were mounted on this second rack, one of which was maintained at a constant temperature of 42.5 ± 2 F. and a constant relative humidity of 50 ± 5 per cent, the other cabinet was maintained at a constant temperature of 128.4 ± 2 F. and a constant relative humidity of 50 ± 2 per cent.

The following equipment was common to both creep racks: calibrated weights and levers for loading the specimens in tension; calibrated mechanical extensometers, a travelling microscope used for measuring the strain indicated by the extensometer; a clock equipped with a counter to record the elapsed time in hours; the necessary equipment to control and maintain the correct temperatures and humidities; and three analytical balances for weighing control specimens at each temperature.

A general view of the apparatus for tests at 42.5 and 128.4 F. is shown in Fig. 2. All creep racks were mounted on concrete slabs *A* which in turn rested on a bed of sand about 10 in. deep. The purpose of the sand was to isolate the creep racks from building vibrations.

The cabinets *B* and *C*, Fig. 2, were constructed in the same manner for both the high and low temperatures. The arrangement used is shown in Fig. 3. Each cabinet consisted of a box insulated with 4 in. of glass fiber. The cabinet was lined with galvanized sheet steel soldered at the joints, and the cover was provided with a rubber seal to prevent water vapor from entering or leaving the cabinet. Tubes *A*, Fig. 3, were provided in the top and bottom of the cabinets to permit passage of the rods used for transmitting load to the specimens. The tubes through which the loading rods passed were large enough to permit alignment of the loading rods and were sealed to prevent passage of water vapor by rubber membranes *B*. Two additional smaller holes were provided in the top of each cabinet to permit weighing of the control specimens.

A triple pane glass observation window (*D* in Fig. 2; *C*, in Fig. 3) was installed in each cabinet cover. A desiccant was placed between the glass panes to prevent condensation of moisture on the inner surfaces.

The high-temperature cabinet was heated by means of an electric strip-heater (*E*, Fig. 3) which extended nearly the full length of the cabinet. The cooling element in the low-temperature cabinet was a eutectic-filled cold plate, *F*, which served as the evaporation chamber in a sulfur dioxide refrigeration system. The eutectic-filled cold plate was used instead of open coils so that temperatures as low as 40 F. could be used with a minimum of disturbance of the relative humidity in the cabinet by condensation on the cooling coils. Both high and low temperatures were controlled by means of thermostats.

Natural convection caused the circulation of the air in the cabinets. This circulation resulted from mounting a transite-board baffle lengthwise of the cabinet, as shown by *D*, Fig. 3. The baffle was so placed as to shield the specimens from radiation from (or to) the heating or cooling element, thus providing a more uniform temperature distribution.

Measurement of the temperature in the cabinets was accomplished by means of three thermocouples mounted as shown in Fig. 3. The thermocouples were located as follows: T.C. 1 in a tray of solution used for humidity control as described below; T.C. 2 in a hole in the bottom grip on a specimen near the center of the cabinet; and T.C. 3 in a hole in the top grip of the same specimen. The thermocouples were mounted in the grips so that cyclic fluctuations in temperature of the air would be averaged out by the large thermal capacity of the grip.

A survey of the temperature variation in the cabinets both with respect to space and time showed the following variations (obtained from readings of a uniformly spaced network of thermocouples): The cyclic variation in temperature of the air in the cabinet at any given point was about 3 F. in the high-temperature and 2 F. in the low-temperature cabinet; the cyclic variation in temperature of the specimen was about 1 F. in both cabinets; the temperature differential over the test section of the specimen was 2 F. in the high-temperature and 4 F. in the low-temperature cabinet; and the variation in temperature from the middle to one end of the cabinet at the height of the test specimens was about 1 F. in the low-temperature and 3 F. in the high-temperature cabinet.

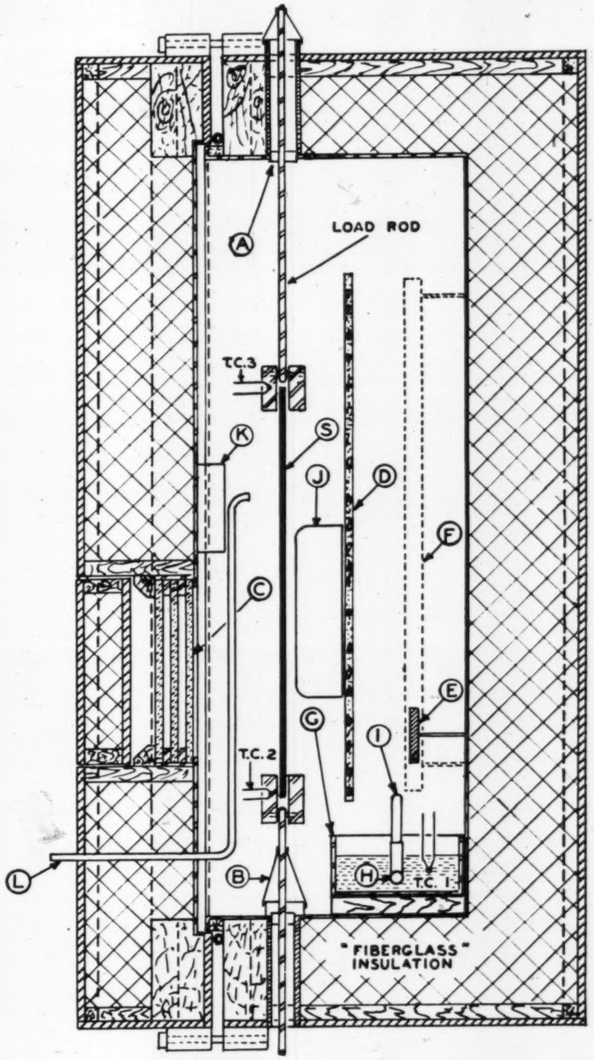


FIG. 3.—Cross-Section of Insulated Cabinet, Elevation.

The control and measurement of the

relative humidity in small test cabinets over the long periods of time necessary for creep tests presented a difficult problem. The method of control of the relative humidity which was finally selected as most suitable is based on the fact that water solutions of many materials (salts, acids, etc.) in given concentrations and at a given temperature produce a fixed relative humidity in the air above the solution when placed in a vapor-tight container long enough for the partial pressure to reach equilibrium.

Of the many solutions available, the one which seemed most suitable was a glycerol-water solution. Sulfuric acid, sodium hydroxide, and salt solutions were not used because of difficulties from corrosion, crystallization (of salts), and control of the concentration.

To control the humidity, a solution of glycerol and water was placed in the tray *G* (Fig. 3) extending the length of the cabinet. The concentration of the solution in each tray was adjusted by withdrawing solution and then adding water if the humidity was too low or glycerol if the humidity was too high. The solutions were withdrawn and added through tube *I* (Fig. 3) extending through the wall of the cabinet and attached to a $\frac{1}{2}$-in. diameter copper tube lying in the bottom of the tray. This tube extended the length of the tray and was closed at both ends. Several small holes were equally spaced along the tube and were of such a diameter as to allow approximately equal amounts of liquid to flow through each when adding liquid. This was done in order to obtain nearly uniform mixing of the new and old liquid along the length of the tray.

The concentration of the solution was measured when necessary by withdrawing a sample and determining the specific gravity with a precision hygrometer.

Control of the relative humidity by this method did not prove as easy as anticipated due to diffusion of water vapor through the rubber seal around the loading rods and the rubber seal between the cover and the cabinet. Water vapor diffused out of the cabinet at 128.4 F. and diffused into the cabinet at 42.5 F. Thus to maintain 50 per cent relative humidity the glycerol-water solution had to absorb or give off water vapor at a rate equal to that at which the water vapor diffused out of or into the cabinet. This process caused changes in the concentration of the solutions, necessitating frequent readjustments. In the high-temperature cabinet the concentration of the solution was approximately the value (76 per cent by volume) required for 50 per cent relative humidity in a vapor-tight cabinet, but in the low-temperature cabinet a concentration of 90 to 95 per cent glycerol was required instead of the 65 per cent for a vapor-tight cabinet.

Of the many techniques for determining relative humidity, most were found to be unsuitable for measurements in small cabinets over long periods of time at the temperatures used. The wet and dry bulb thermometer technique introduced too much water vapor in the cabinet, causing a change in relative humidity of 5 to 10 per cent while taking a reading. Hair-type relative humidity indicators did not repeat calibration when the temperature was changed from either 128.4 F. or 42.5 F. to room temperature and back again.

Other methods considered did not have sufficient precision or lacked adequate stability for long-time tests. The method finally employed was a direct quantitative analysis of a sample of the air in the cabinets. A sample of air was withdrawn through tube *L* (Fig. 3) whose inlet was about in the center of the space occupied by the test specimens. The sample of air was drawn through three U-tubes *E*, Fig. 2 in series containing magnesium perchlorate as a hygroscopic agent, and through a wet test gas meter *F* (Fig. 2) by means of a vacuum pump *G* (Fig. 2). In the high-temperature cabinet it was necessary to heat the portion of the sampling tube outside the cabinet to prevent the moisture in the sample of air from condensing on the walls of the sampling tube.

The difference in weight of the three U-tubes before and after the metered sample of air was drawn through them, indicated the amount of water vapor in the sample. From the weight of water in the sample, the volume of air sampled and the temperature of the air in the cabinet the per cent relative humidity was calculated.

A control specimen of each material was supported freely in the cabinet by a system of links which extended through the top of the cabinet. The linkage was attached to the weighing beam of an analytical balance *H* (Fig. 2) so that the change in weight of the specimen could be determined during the testing period.

The apparatus used for measuring the creep is shown in Fig. 4. In this figure, specimen *A* was subjected to an axial tensile load through rod *B*. The specimen was held by grips *C* which contained a hook-and-eye type of swivel joint. Such a joint was provided in order to minimize the possibility of loading the specimen eccentrically.

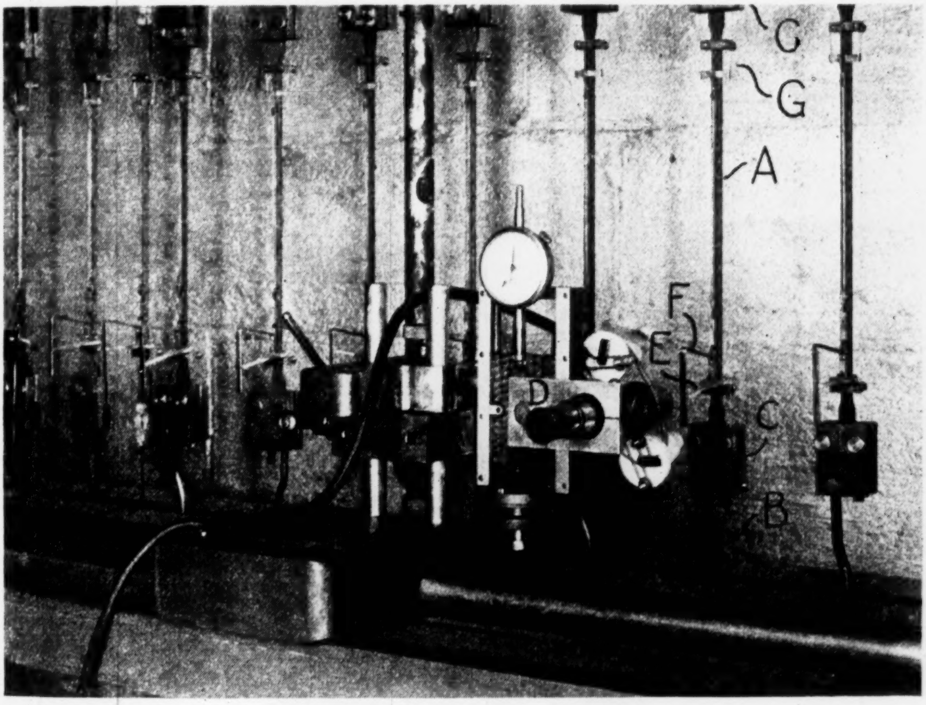


FIG. 4.—Apparatus Used for Making Creep Measurements.

The extensometer consisted of a lever type instrument with a travelling microscope (*D*, Fig. 4) for measuring the displacement between reference marks on the end of the lever *E*, and on the stationary arm *F*. The lever ratio was 10 to 1. One end of the lever was forked and fastened by pivots to the lower clamp attached to the specimen. The axis of this pivot passed through the centroid of the cross-section of the specimen (the pin itself did not to through the specimen). Thus the strain measured by this instrument was the average strain in the specimen. The fulcrum of the lever was pivoted to a rod, the other end of which was fastened to the upper clamp on the specimen. A spring clip *G* (Fig. 4) was used to attach this rod to the upper clamp so that the extensometer could be left on the specimen during fracture, if necessary, without damage to the instrument.

TABLE III.—SUMMARY OF CREEP TESTS.

Material	Temperature, deg. Fahr.	Relative Humidity, per cent	Stress, psi.	Total Creep at 800 hr. Corrected for shrinkage, per cent	Rate of Creep at 800 hr., Corrected for Shrinkage, per cent	20 sec. "Elastic" Strain, per cent	"Elastic" Modulus from Creep Tests, psi.
Canvas laminate.....	42.5	50	0	0	0	0	910 000
			1 300	0.19	0.17	0.146	
			2 600	0.40	1.78	0.299	
			3 800	0.69	3.92	0.448	
			5 100	1.19	7.24	0.689	
Canvas laminate.....	77.0	50	0	0	0	0	836 000
			600	0.095	1.50	0.065	
			1 200	0.200	2.17	0.142	
			1 800	0.321	3.84	0.218	
			2 400	0.485	4.94	0.307	
			3 000	0.635	7.17	0.394	
			3 600	0.853	8.18	0.481	
			4 200	1.115	10.02	0.610	
Canvas laminate.....	128.4	50	0	0	0	0	750 000
			1 000	0.268	6.40	0.132	
			1 900	0.555	9.84	0.268	
			2 900	1.010	16.77	0.464	
			3 800	1.486	20.10	0.660	
Rayon laminate......	42.5	50	0	0	0	0	1 900 000
			3 600	0.266	0.53	0.192	
			7 200	0.692	5.09	0.402	
			10 800	1.362	7.39	0.675	
			14 400	2.105	9.04	1.160	
Rayon laminate......	77.0	50	0	0	0	0	1 700 000
			1 900	0.175	1.27	0.120	
			3 900	0.395	2.72	0.222	
			5 800	0.687	4.78	0.351	
			7 800	1.045	6.43	0.520	
			9 700	1.405	7.77	0.720	
			11 600	1.808	9.06	0.965	
			13 600	2.285	9.72	1.320	
Rayon laminate......	128.4	50	0	0	0	0	1 320 000
			2 600	0.390	4.97	0.196	
			5 200	0.824	6.88	0.402	
			7 800	1.350	8.40	0.726	
			10 400	1.934	10.13	1.170	

Material	Temperature, deg. Fahr.	Constants for Hyperbolic-Sine Equation		Activation Energy, $\triangle H$, cal. per mole	Activation Entropy, $\triangle S$, cal. per deg. K. per mole	qAl, (Å)^3
		ν_0, in. per in. per 10^7hr.	σ_0, psi.			
Canvas laminate......	42.5	0.87	1 800	16 000	−49.6	622
	77.0	5.9	3 100	16 000	−49.6	386
	128.4	68.	12 500	16 000	−49.6	105
Rayon laminate......	42.5	....	....	....	....	....
	77.0	6.9	10 100	....	....	....
	128.4	....	....	....	....	....

Each instrument was calibrated against a micrometer screw before use.

Spring-loaded flat clamps were used to attach the extensometer to the specimen instead of pointed screws to avoid early fracture which might result from the indentation of screws. A gage length of 10 in. was used for the majority of specimens tested, but a 4-in. gage length was used on specimens subjected to high stresses where a large amount of strain was expected. A track along the front of the creep rack made it possible to move the microscope from specimen to specimen quickly.

TEST PROCEDURE

All creep specimens for both laminates were subjected to constant tension loads which would produce values of stress evenly spaced from zero to a maximum. The maximum stress was based on the results of preliminary creep tests. The values of stress used are given in Table III.

The preliminary creep tests were started at a stress of about 60 per cent of the tensile strength of the material, and the stress was increased in steps of about 6 per cent every 24 hr. until the specimen failed. The maximum stress to be used in the creep tests was then made equal to 80 per cent of the highest stress which the preliminary creep specimen sustained for 24 hr. The reason for the preliminary creep tests was to select as high a maximum stress as possible without having a specimen fracture within 1000 hr. However, the rayon specimen subjected to the highest stress at 77 F. did fail at about 900 hr.

One specimen of each laminate at each temperature was maintained at zero stress in order to determine the magnitude of the contraction (shrinkage) or expansion due to gradual changes in moisture content or other aging phenomena.

All tests were started simultaneously as nearly as possible. The tests at 77 F. were all started during the same day. Two weeks later the tests at the high and low temperatures were started during one day. The tests at 77 F. were conducted in the room in which the specimens were stored. However, the specimens tested at 42.5 F. and 128.4 F. were removed from the room in which they were stored and transferred to the test cabinets just prior to start of these tests. During the time that the specimens were being mounted in the cabinets and minor adjustments were being made on the apparatus, the specimens were subjected to a temperature of about 85 F. and a relative humidity of about 80 per cent.

Operation of the cabinets was started 6 hr. after the specimens were removed from the room in which they were stored. About 2½ hr. were required for the cabinets to reach the desired temperature and about 39 hr. elapsed before the relative humidity had been adjusted to the desired range. The creep tests in the cabinets were started about 46 hr. after operation of the cabinets commenced.

In starting the creep tests at all three temperatures, the initial readings of the extensometers and the weight of the control specimen were determined first. Then the load was applied quickly but very gently to a specimen, the extensometer was immediately read again, and the time was recorded. This process was repeated for each specimen. Subsequent readings of strain and time were taken at about the following intervals: 0.1, 0.2, 0.5, 0.7, 1, 2, 3, 5, 7, 10, 20 hr.; then every 24 hr. to 500 hr.; then every 48 hr. to 1000 hr.

As soon as the tests in the high- and low-temperature cabinets were started, the humidity, temperature and weight of the control specimen were determined. This information was recorded thereafter at intervals of 2 to 4 days.

RESULTS

The creep in per cent as a function of time up to 1000 hr. and at several different values of stress is shown in Figs. 5 to 10 for each material at each of the three temperatures. To aid in the interpretation of the data, the relative humidity, the weight of the control specimen, and the change in length of a specimen carrying no load are also shown.

It was observed that the shape of the creep curves was essentially the same at all loads, all temperatures, and for both materials. The rate of creep was rapid at the beginning of the test and decreased as time progressed. The lowest curve plotted in each case was taken from data obtained with a specimen which carried no load, that is, zero stress. The specimen carrying no load showed shrinkage at temperatures of 128.4 and 77 F. and showed a gradual extension with time at 42.5 F. for both laminates. The amount of the shrinkage was about the same for

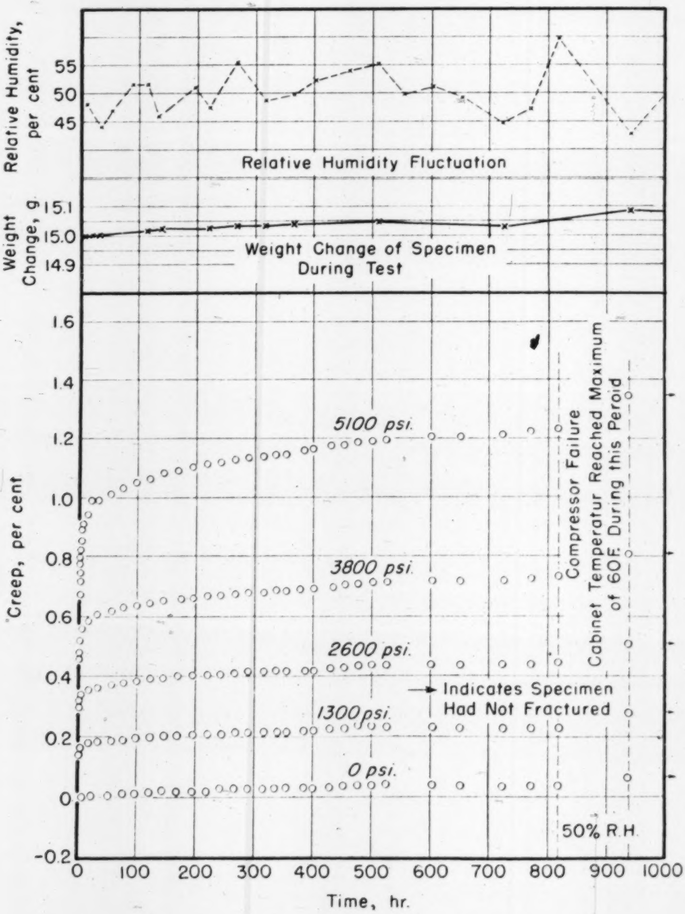


FIG. 5.—Creep *versus* Time for Tension Creep Tests at 42.5 F. of Low-Pressure Canvas Laminate.

both laminates—0.04 per cent at 128.4 F. and 0.01 per cent at 77 F. The extension was 0.03 and 0.05 per cent for the rayon and canvas laminates respectively at 42.5 F.

A change in weight of the control specimens occurred simultaneously with the change in dimensions. With one exception, an increase in length of the specimen carrying no load was accompanied by an increase in weight of the control specimen and *vice versa.* The weight of the canvas laminate, however, was nearly constant during the test period. Examination of Figs. 5 to 10 reveals the effect of changes in the relative humidity. An increase in the relative humidity caused an increase in the amount of creep (strain) that took place in a given time interval and *vice versa.* The amount of the change in creep was approximately the same for specimens at all stresses for a given temperature and material.

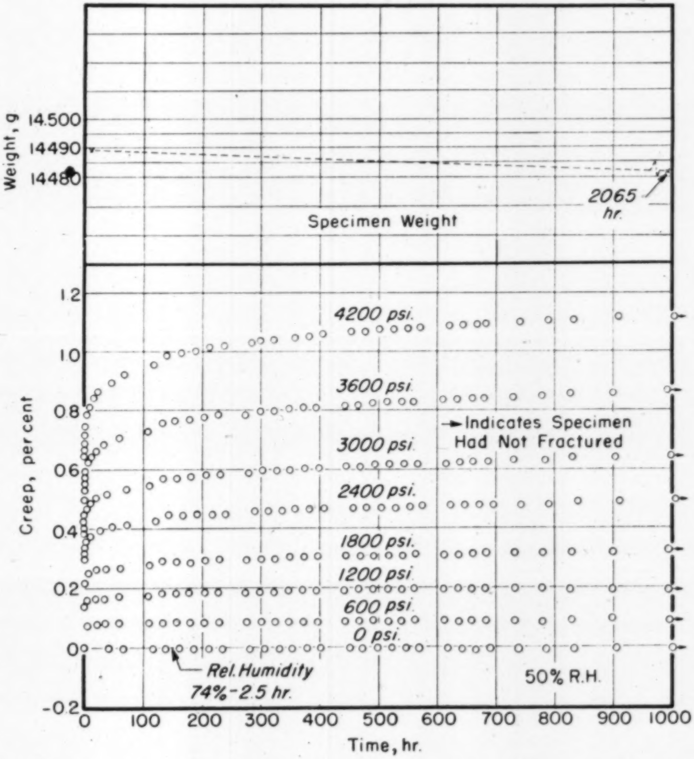


FIG. 6.—Creep *versus* Time for Tension Creep Tests at 77 F. of Low-Pressure Canvas Laminate.

The elastic portion of the strain occurring during a creep test is defined in this paper as the total strain measured in the specimen at the time at which the load had been fully applied to the specimen for 20 sec Since a reading was seldom obtained at exactly 20 sec. the 20-sec. "elastic" strain was determined by plotting the readings obtained during the first hour of tests on log-log paper. A straight line was then drawn through the plotted points for each stress, and the strain corresponding to a time of 20 sec. was read from the straight line. This technique was justified because of the fact brought out in the 1944 paper by Findley (15) that creep data from materials of this type yield nearly linear curves when plotted on log-log coordinate paper.

The values of 20-sec. "elastic" strain are given in Table III, and are plotted as functions of stress for the three temperatures in Fig. 11 for the canvas laminate and in Fig. 12 for the rayon laminate The data at each temperature form smooth curves similar to the stress-strain curves reported in the paper by Findley and Worley (8) for the laminates except that the coordinates are reversed. In fact, the modulus of elasticity obtained at 77 F. from Fig. 11 is only about 3 per cent less than the elastic modulus obtained from tension tests of the canvas

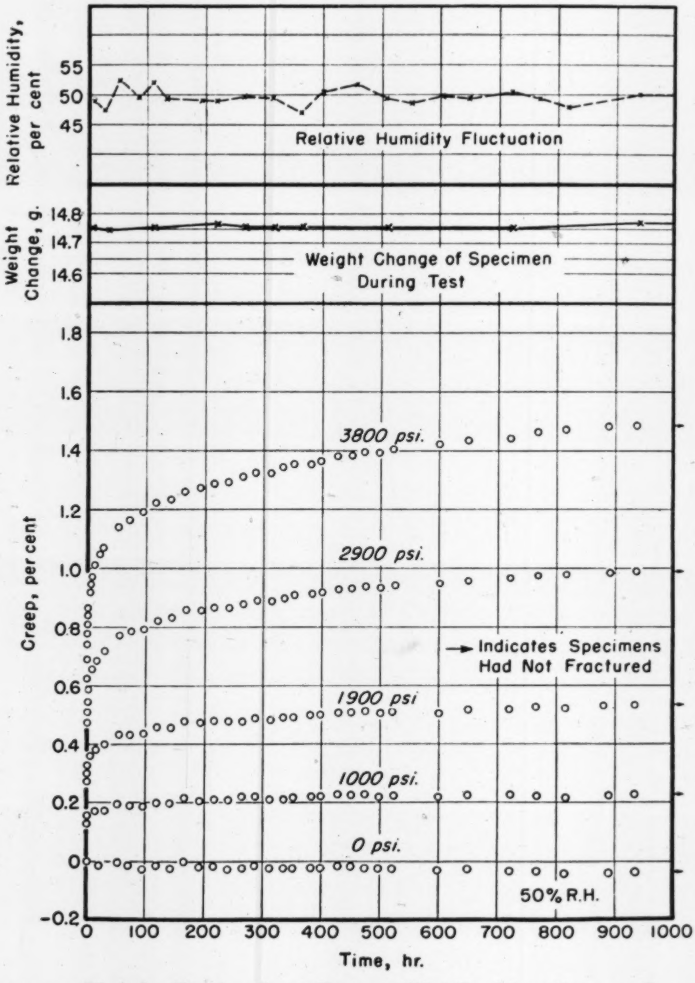


FIG. 7.—Creep *versus* Time for Tension Creep Tests at 128.4 F. of Low Pressure Canvas Laminate

laminate, and the modulus obtained at 77 F. from Fig. 12 is about 10 per cent higher than the elastic modulus obtained from tension tests of the rayon laminate. Figure 13 shows the effect of temperature on the "elastic" strain for two values of stress for each laminate.

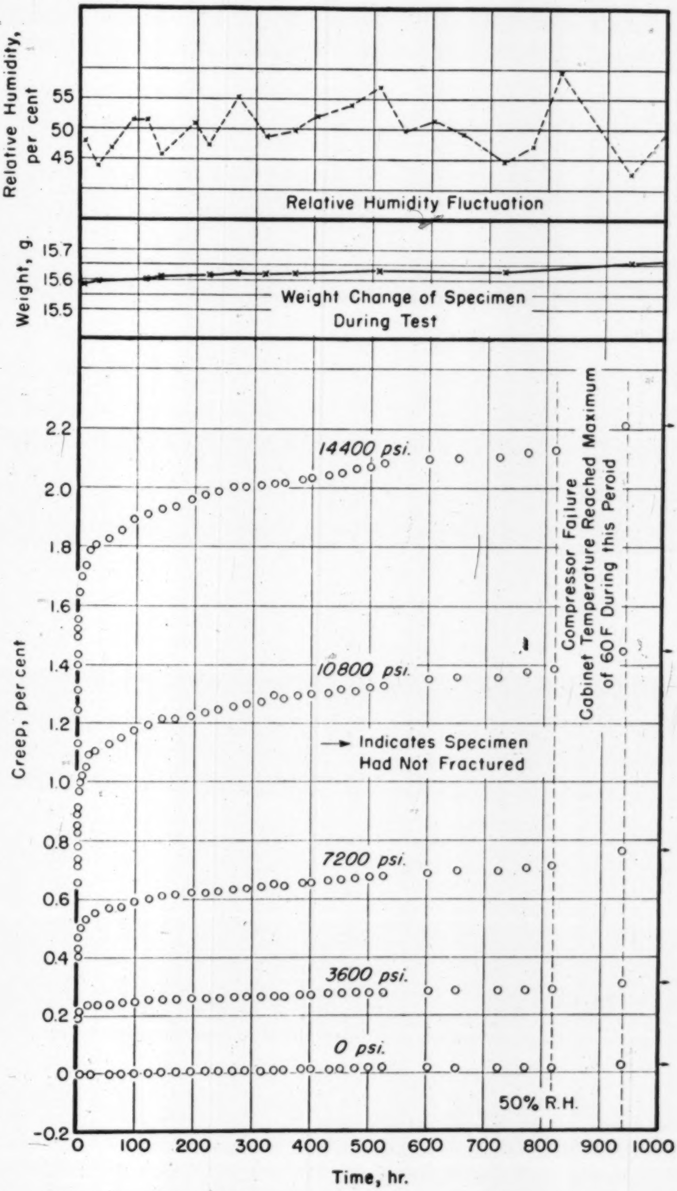


FIG. 8.—Creep *versus* Time for Tension Creep Tests at 46.5 F. of Rayon-Cotton Laminate.

The total creep (including the elastic strain) was measured for all specimens at 800 hr. These data were then corrected

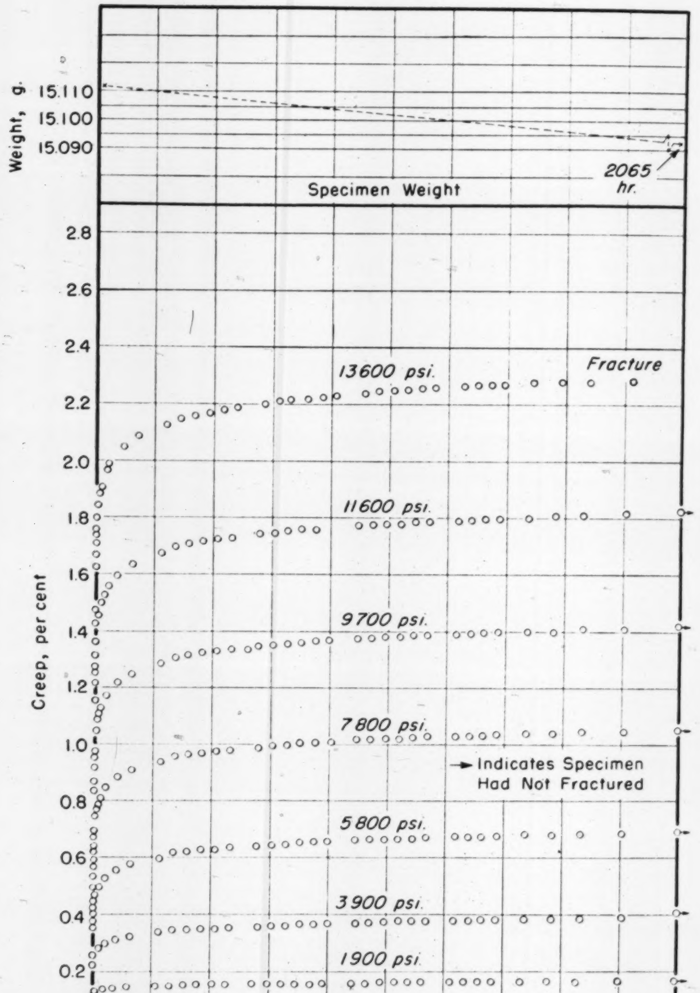


FIG. 9.—Creep *versus* Time for Tension Creep Tests at 77 F. of Rayon-cotton Laminate.

for the change in length of the specimens having zero load by subtracting the algebraic value of the change in length of this specimen from the total creep. The corrected values of total creep are shown in Table III and are plotted as functions of stress for the three temperatures in Fig. 14 for the canvas laminate and in Fig. 15 for the rayon laminate.

In Fig. 16 the effect of temperature

on the total creep at three different values of stress is shown for each laminate. The relation between the total creep at 800 hr. and the temperature appears to be nearly linear within the small range of temperatures studied. It is evident from Fig. 16 that the rayon laminate may be subjected to about twice as much stress as the canvas laminate to produce the same total creep in 800 hr. for any temperature within the range of temperatures studied.

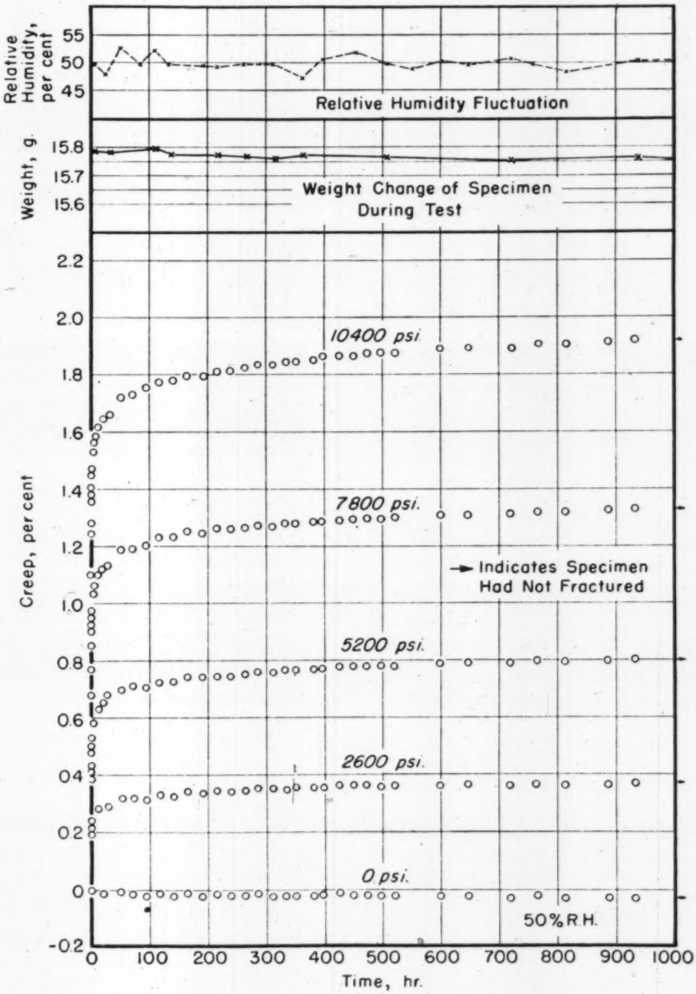


FIG. 10.—Creep *versus* Time for Tension Creep Tests at 128.4 F. of Rayon-cotton Laminate.

The rate of creep did not remain constant throughout the time of testing but decreased rapidly at first and then more gradually. In order to evaluate the effect of stress on the different rates of creep, therefore, it was necessary to

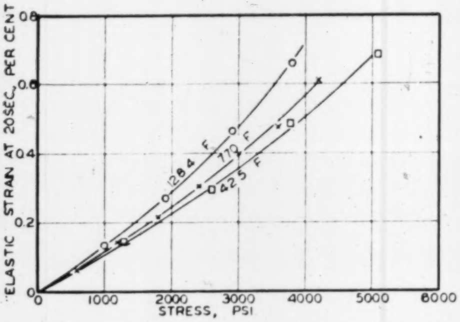


Fig. 11.—The Effect of Stress at Three Different Temperatures on the 20-sec. "Elastic" Strain of Low-Pressure Canvas Laminate.

determine the rate of creep at some definite time. The rate of creep was determined at 800 hr. since the refrigeration compressor for the low-temperature tests failed at that time causing an increase in temperature. The rate of creep was determined by measuring the slope of the creep-time curves at 800 hr. When determining the slopes from the creep-time curves, particularly for the tests at 42.5 F. it was necessary to consider the effect of fluctuations in relative humidity. Data taken during time intervals when

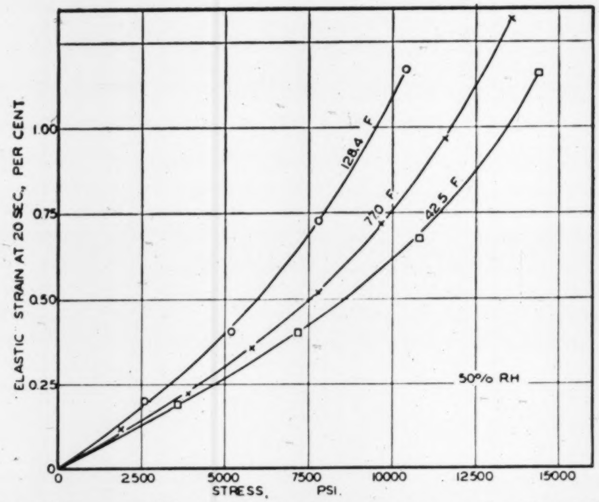


Fig. 12.—The Effect of Stress at Three Different Temperatures on the 20-sec. "Elastic" Strain of Rayon-cotton Laminate.

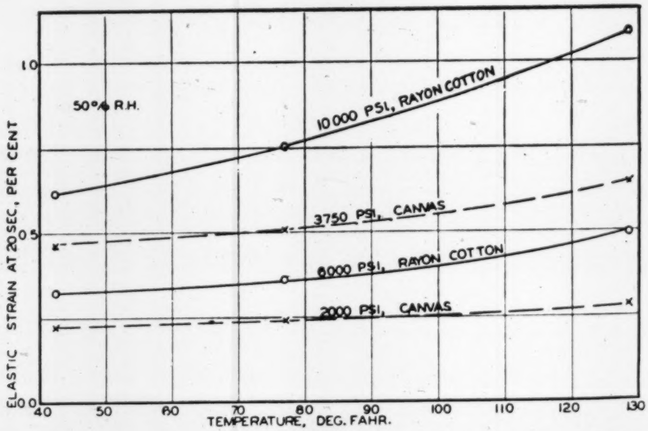


Fig. 13.—The Effect of Temperature on the 20-sec. "Elastic" Strain of Low-Pressure Canvas and Rayon-cotton Laminates at Two Values of Stress for Each Material.

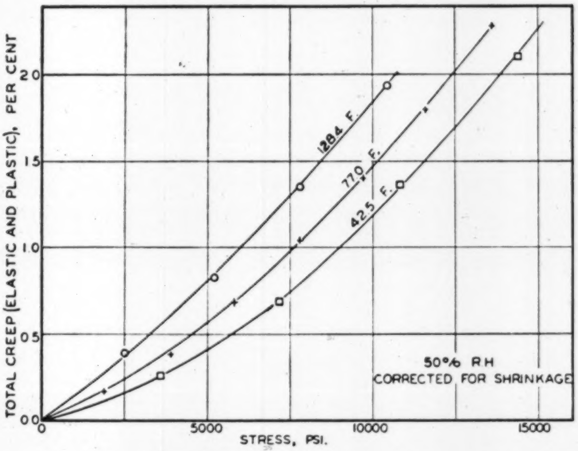


FIG. 14.—The Effect of Stress at Three Different Temperatures on the Total Creep of Low-Pressure Canvas Laminate at 800 hr.

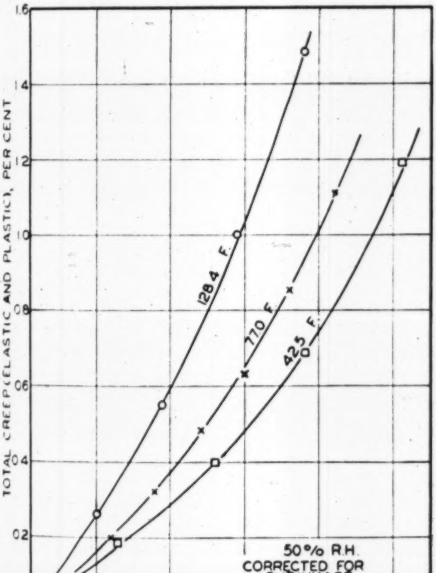


FIG. 15.—The Effect of Stress at Three Different Temperatures on the Total Creep of Rayon-cotton Laminate at 800 hr.

the relative humidity was nearly 50 per cent were given more weight in determining the slopes than data taken during times when the relative humidity was too high or too low.

The rate of creep at 800 hr. is given in Table III and is plotted for each temperature in Fig. 17 for the canvas laminate and in Fig. 18 for the rayon laminate. It is evident that the rate of creep increases with the stress and the temperature. The effect of temperature on the rate of creep is best shown in Fig. 19 in which the rate of creep for two different stresses is plotted as a function of temperature for both laminates. This figure shows that the rate of creep of the canvas laminate increased much more rapidly with increasing temperature than the rate of creep of the rayon laminate and that the rate of creep for a given stress was substantially less for the rayon than the cotton laminate within the temperatures studied.

In the paper by Findley and Worley (8) additional data are given on the creep of these two laminates at 77 F. The creep curves in this reference extend to 3000 hr., and the rate of creep at 1000 hr. is shown.

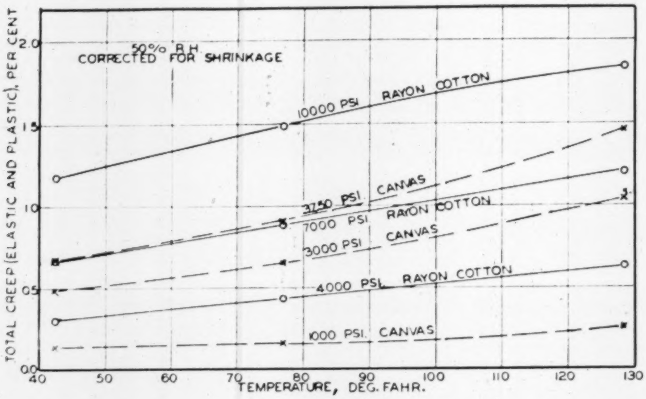


FIG. 16.—The Effect of Temperature on the Total Creep at 800 hr. of Low-Pressure Canvas and Rayon-cotton Laminates at Three Values of Stress for each Material.

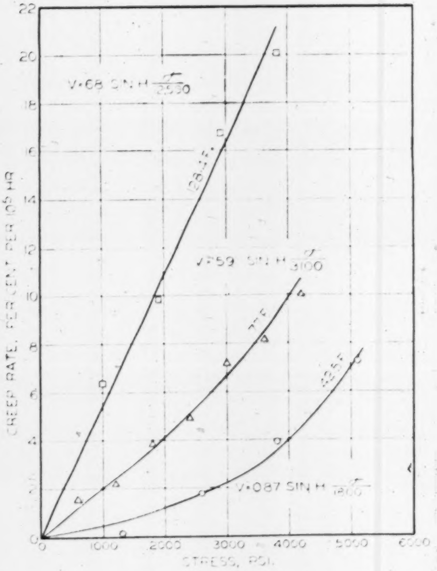


FIG. 17.—The Effect of Stress at Three Different Temperatures on the Creep Rate of Low-Pressure Canvas Laminate at 800 hr.

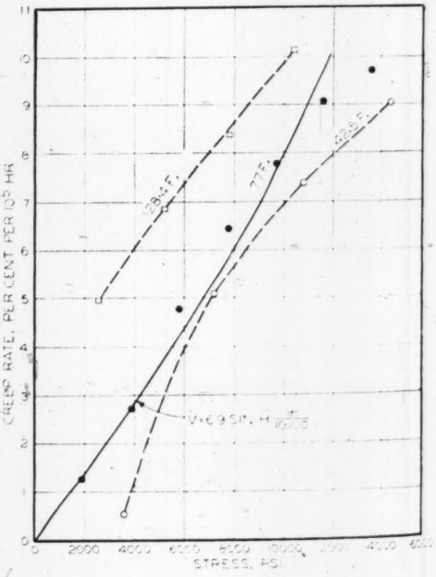


FIG. 18.—The Effect of Stress at Three Different Temperatures on the Creep Rate of Rayon-cotton Laminate at 800 hr.

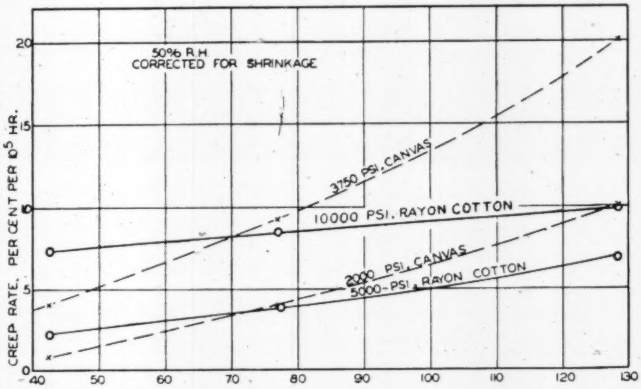


FIG. 19.—The Effect of Temperature on the Rate of Creep at 800 hr. for Each Material at Two Values of Stress.

ANALYSIS OF DATA BY THE ACTIVATION ENERGY THEORY

The activation energy theory, as proposed by Eyring (**16**) for chemical reaction rates and for diffusion, has been applied to the problem of the creep characteristics of metals by Kanter (**17**), Kauzmann (**18**), Dushman[4] (**19**), and others and to the creep of thermoplastics by Kistler (**10**). The theory was usually applied by these investigators to conditions in which creep occurred at constant rate. Most plastics do not appear to creep at a constant rate within the time intervals that have been investigated. However, as suggested in reference (**15**) the activation-energy theory may be applicable to the creep rates existing at a given time—such as 800 hr. in the present series of tests. The possibility has been investigated as follows:

The activation energy equation for creep rates of metals as suggested by Kauzmann for the general case where any range of values of the quantity $\frac{qAl\sigma}{2kT}$ may be encountered is

$$v = \frac{4}{3} \frac{\lambda}{L} \frac{k}{h} T e^{\Delta S/R} e^{-\Delta H/RT} \operatorname{Sinh} \frac{qA\ell\sigma}{2kT} \quad . . (1)$$

where:

v = the rate of creep, inch per inch per second,

T = the absolute temperature, in degrees Kelvin,

σ = the tensile stress, in dynes per square centimeters,

k = the Boltzmann constant, 1.3805 × 10^{-16} erg per degrees Kelvin,

h = the Planck constant, 6.624 × 10^{-27} erg seconds,

R = the gas constant per mole, 1.986 cal. per deg. Kelvin per mole,

A = the area of a unit of flow in the plane in which the shear stress acts in square centimeters,

λ = the distance in the direction of the shear stress moved by the units of flow relative to one another in a single place change or jump in centimeters,

l = the distance through which the shear stress acts in carrying the unit of flow from the normal to the activated state,

L = the distance between the layers of units of flow in centimeters,

---

[4] Contains a brief summary of the theory as developed for creep of metals by Kauzmann.

q = a stress concentration factor,
ΔS = the entropy of activation per mole in calories per degree Kelvin per mole, and
ΔH = the energy of activation per mole, calories per mole.

An empirical expression of this form has been proposed by Nadai (20) and tested by McVetty (21) to express the relation between stress and creep rate of metals. The empirical equation may be expressed as:

$$v = v_o \operatorname{Sinh} \frac{\sigma}{\sigma_o} \qquad (2)$$

where v_o and σ_o are constants.

It is easily shown that the hyperbolic sine term in either Eq. 1 or 2 may be simplified when we are concerned with large values or very small values of the variable, $\frac{qAl\sigma}{2kT}$ or $\frac{\sigma}{\sigma_o}$, since Sinh $x \approx \frac{e^x}{2}$ for large values of x and Sinh $x \approx x$ for small values of x.

The first simplifying condition has been used with success by Kauzmann and Dushman in calculating values of the energy and entropy of activation for creep tests of several metals. Such a relationship has also long been used as an interpolation formula for creep rates of metals. In this application the log of creep rate is plotted as a function of stress, and straight lines result.

The second simplifying condition, small values of x, probably has little utility in engineering materials. While it would serve well as an interpolation formula within its limiting conditions, small values of x would make it impossible to separate v_o and σ_o in Eq. 2 since they merge into one constant and would also make impossible the calculation of ΔS, ΔH and qAl from Eq. 1.

The calculation of the activation energy from the creep rates given in Table III was tried for the first simplifying condition by plotting the log of creep rate versus stress. The marked curvature of the resulting constant temperature lines indicated that the values of x, that is $\frac{qAl\sigma}{2kT}$, were too small to use this simplification. Thus it was necessary to use the hyperbolic sine function. Values of v_o and σ_o, Eq. 2, were selected which would make Eq. 2 best fit the data. The nomograph prepared by McVetty (21) was used to approximate the values of v_o and σ_o and the final values were determined by trial. The values of v_o and σ_o selected are given in Table III. Agreement between the equations selected and the experimental data is shown for the canvas laminate in Fig. 17 and for one of the curves of the rayon laminate in Fig. 18. The hyperbolic sine curves are shown as solid lines.

Scatter in the data for the creep rates of the rayon laminate at the high and low temperature precluded any further study of these data; at least these data do not even approximate a hyperbolic sine relation. Whether the disagreement represents scatter in the test results or not is, of course, not known; but it is recognized that creep rates are difficult to measure by determining the tangent to a curve.

A better technique, perhaps, would be to fit the entire creep *versus* time data to an equation such as:

$$\epsilon = \epsilon_o + mt^n, \qquad (3)$$

where:
ϵ = the creep,
t = the time, and
ϵ_o, m are constants.

as described in reference (15). The creep rate at the given time is then obtained by differentiating the equation, giving

$$v = \frac{d\epsilon}{dt} = mnt^{n-1}, \qquad (4)$$

and substituting the desired value of time, t.

This procedure is being applied to the creep data reported herein for presentation at a later date.

From values of v_o, σ_o and the absolute temperature T it was possible to calculate the quantities ΔS, ΔH, and qAl by the following manipulations:

A comparison of Eqs. 1 and 2 yields the following identities:

$$v_o = \frac{4}{3}\frac{\lambda}{L}\frac{k}{h}\,T e^{\Delta S/R}\, e^{-\Delta H/RT} \ldots\ldots (5)$$

and

$$\frac{1}{\sigma_0} = \frac{qA\ell}{2kT} \ldots\ldots\ldots\ldots (6)$$

Since λ and L are presumed to be the same order of magnitude of interatomic distances the approximation $\frac{4}{3}\frac{\lambda}{L} \approx 1$ may be introduced.

Equation 5 may then be written as

$$\log_{10}\left(\frac{v_o}{T}\right) = \log_{10}\frac{k}{h} + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303\mathrm{R}}\left(\frac{1}{T}\right) \ldots (7)$$

where 2.303 is the conversion factor from natural to ordinary logarithms. Thus values of $\log_{10}\left(\frac{v_o}{T}\right)$ *versus* $\left(\frac{1}{T}\right)$ should plot as a straight line. From the slope and intercept of this straight line, the values of ΔH and ΔS respectively may be calculated. Figure 20 shows such a diagram of $\frac{v_o}{T}$ *versus* $\frac{1}{T}$ for the canvas laminate constructed with the values of v_o used in drawing the solid lines shown in Fig. 17.

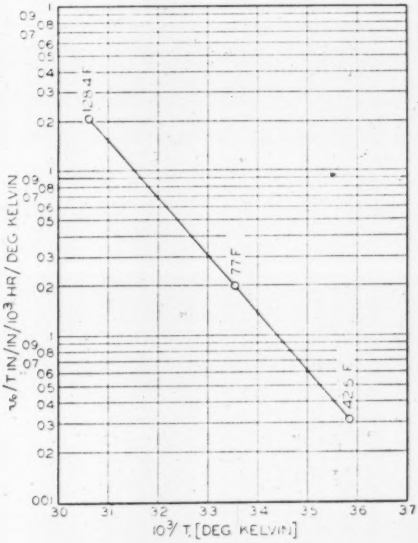


FIG. 20. A Plot of $\frac{v_o}{T}$ *versus* $\frac{1}{T}$ for Low-Pressure Canvas Laminate.

The left-hand point in Fig. 20 corresponds to the curve for 128.4 F. in Fig. 17. Since the curve for 128.4 F. is practically a straight line it must fit the condition that $\sinh\frac{\sigma}{\sigma_o} \approx \frac{\sigma}{\sigma_o}$. Thus $v \approx \frac{v_o}{\sigma_o}\sigma = c\sigma$ so that v_o and σ_o can be changed over wide limits (as long as their ratio, C, is held constant) without affecting the degree of fit between the curve in Fig. 17 for 128.4 F. and the data. Because there is no accurate way to evaluate v_o and σ_o independently for this condition, the point on the curve in Fig. 20 corresponding to 128.4 F. is not accurately known. For this reason the value of v_o was chosen so that it fell on a straight line passing through the other two points shown in Fig. 20. The values of v_o for the other temperatures are, however, prescribed within rather narrow limits by the necessity that Eq. 2 fit the data in Fig. 17.

The value obtained for the energy of activation ΔH was 16,000 cal. per mole and the value of the entropy of activation was -49.6 cal. per deg. Kelvin per mole. These values are the same order of magnitude as those for creep of metals as determined by Kauzmann (**18**) and Dushman (**19**). In fact they are almost identical with the values calculated by Kauzmann from the creep data on a special grade of zinc brass tested by Orowan (**22**). The fact that the entropy of activation is negative does not seem reasonable but agrees with the results of most of the metals studied by Kauzmann and Dushman. These two investigators have proposed certain explanations for the negative sign which, however, are not very convincing either to them or to the authors.

The value of the quantity qAl may be determined from Eq. 6 for each temperature by substituting the known values of σ_o and T. The values obtained for qAl, which range from 105 to 622 $(\text{Å})^3$, are of the same order of magnitude of some of the metals studied by Kauzmann but decrease with increasing temperature whereas 12 out of the 14 metals studied by Kauzmann showed an increase in qAl with temperature.

CONCLUSIONS

1. The relation between the total creep at 800 hr. and the temperature appeares to be nearly linear within the temperature range from 42.5 to 128.4 F. for both laminates studied.

2. The canvas laminate will withstand only about one half as much stress as the rayon laminate without suffering a larger total creep in 800 hr. than the rayon laminate within the range of temperature tested.

3. The rate of creep of the canvas laminate increased much more rapidly with increasing temperature than the rate of creep of the rayon laminate.

4. For a given stress the rate of creep was substantially less for the rayon than the canvas laminate within the range of temperature tested.

5. The rates of creep obtained at 800 hr. for the canvas laminate agree fairly well with the activation energy theory, but the rates of creep obtained for the rayon laminate do not.

6. The quantities obtained from the activation energy theory for the canvas laminate are: energy of activation 16,000 cal. per mole; entropy of activation -49.6 cal. per deg. Kelvin per mole, and product qAl 105 to 622 $(\text{Å})^3$.

7. The values of energy of activation, entropy of activation, and product qAl agree both in magnitude and sign with values reported for the creep of metals.

Acknowledgments:

The authors wish to thank the National Advisory Committee for Aeronautics for financial assistance in connection with the tests at 77 F. and for permission to quote the information given in Table I. We are also grateful to Synthane Corporation and Formica Insulation Co. which supplied the laminates used in these tests.

The project covered by this paper was part of the work of the Engineering Experiment Station of the University of Illinois, of which M. L. Enger is Director, in the Department of Theoretical and Applied Mechanics, of which F. B. Seely is Head. The authors are indebted to F. B. Seely for criticism of the paper and to R. L. Sutherland for checking the calculations.

A portion of the project reported was conducted as a thesis for the degree of Master of Science in Theoretical and Applied Mechanics at the University of Illinois by C. H. Adams.

REFERENCES

(1) H. Leaderman, "Creep, Elastic Hysteresis and Damping in Bakelite Under Torsion," *Journal of Applied Mechanics*, June, 1939, p. A-79.

(2) H. Perkuhn, "The Creep of Laminated Synthetic Resin Plastics," NACA Technical Memo. No. 995 (1941).

(3) W. N. Findley, "Mechanical Tests of Cellulose Acetate—Part II on Creep," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 914 (1942).

(4) W. N. Findley, "Mechanical Tests of Macerated Phenolic Molding Material," NACA Advance Restricted Report 3F19, Wartime Report W99, June, 1943.

(5) B. Chasman, "Creep and Time-Fracture Strength of Plastics Under Tensile Stresses," *Modern Plastics*, Vol. 21, No. 6, p. 145, (1944).

(6) W. N. Findley and W. J. Worley, "Short-time Static Tests and Creep Tests of a Paper Laminated Plastic," *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 949 (1944).

(7) Joseph Marin, "Static and Dynamic Creep Properties of Laminate Plastics for Various Types of Stresses," National Advisory Committee for Aeronautics, Technical Note 1105, (1947).

(8) W. N. Findley and W. J. Worley, "Mechanical Properties of Five Laminated Plastics" National Advisory Committee for Aeronautics, Tech. Note. 1560, (1948).

(9) W. F. Bartoe, "Service Temperature Flow Characteristics of Thermoplastics," *Mechanical Engineering*, Vol. 61, No. 12, December, 1939, p. 892.

(10) S. S. Kistler, "The Thermoplastic Behavior of Linear and Three Dimensional Polymers," *Journal of Applied Physics*, Vol. 11, p. 769 (1940).

(11) J. Delmonte, W. Dewar, "Factors Influencing Creep and Cold Flow of Plastics," ASTM BULLETIN, No. 112, October, 1941, p. 35.

(12) R. Burns, "Deformation Under Load of Rigid Plastics," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 1194 (1943).

(13) D. Telfair, T. S. Carswell, H. K. Nason, "Creep Properties of Molded Phenolic Plastics," *Modern Plastics*, Vol. 21, No. 6, February, 1944, p. 137.

(14) W. J. Gailus and David Telfair, "Creep Properties of Molded Phenolic Plastics at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 67, p. 253 (1945).

(15) W. N. Findley, "Creep Characteristics of Plastics," *Symposium on Plastics*, Am. Soc. Testing Mats., Philadelphia, Pa., p. 118 (1944).

(16) H. Eyring, "Viscosity, Plasticity, and Diffusion as Examples of Absolute Reaction Rates," *Journal of Chemical Physics*, Vol. 4, p. 283 (1936).

(17) J. J. Kanter, "The Problem of the Temperature Coefficient of Tensile Creep Rate," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 131, p. 385 (1938).

(18) W. Kauzmann, "Flow of Solid Metals from the Standpoint of the Chemical-Rate Theory," *Transactions*, Am. Inst. of Mining and Metallurgical Engrs., Inst. of Metals Division, Vol. 143, p. 57 (1941).

(19) S. Dushman, L. W. Dunbar, H. Huthsteiner, "Creep of Metals," *Journal of Applied Physics*, Vol. 15, No. 2, p. 108 (1944).

(20) A. Nadai, "The Influence of Time Upon Creep. The Hyperbolic Sine Creep Law," Stephen Timoshenko Anniversary Volume, The Macmillan Co., New York, N. Y. (1938).
Also: "The Creep of Metals Under Various Stress Conditions," Theodore von Kármán Anniversary Volume, Contributions to Applied Mechanics and Related Subjects (1941).

(21) P. G. McVetty, "Creep of Metals at Elevated Temperatures—The Hyperbolic Sine Relation Between Stress and Creep Rate," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, No. 7, October 1943, p. 761.

(22) E. Orowan, "Zur Kristallplastizität, Tieftemperaturplastizität und Beckersche Formel," Zeitschrift für Physik, Vol. 89, p. 605 (1934). Also Vol. 97, p. 573 (1935); Vol. 98, p. 382 (1936).

THE DETERMINATION OF LIMITS FOR THE CONTROL OF PLACEMENT MOISTURE IN HIGH ROLLED-EARTH DAMS*

By W. G. Holtz[1]

Synopsis

The general concept for the control of rolled-earth fill construction has been based upon the premise that the most desirable placement condition for the soil is the condition corresponding to the peak point of the moisture-density (compaction) curve. This criterion is, in many cases, unsatisfactory for high earth dams. When impervious materials having relatively high optimum moisture contents are used in high earth dams, it has been found that the stability of the structure is greatly reduced by the development of pore pressures in soils placed near the optimum condition. This reduction in stability takes place when the mass consolidates as it is loaded during construction, because the developed pore pressure causes a reduction of effective contact pressure between the particles. On the other hand, if the materials are placed too dry, the mass will consolidate excessively when the dam becomes saturated. This paper describes the laboratory tests and analyses used to establish moisture control limits so that neither of these undesirable conditions will exist.

Since the advent of procedures for controlling moisture and compaction in the construction of rolled-earth fills, together with the concepts relative to the significance of optimum moisture content and maximum dry density, the general field control procedure has been based upon the concept that the most desirable condition for the placement of materials in earth fills is the condition corresponding to the peak point of the moisture-density (compaction) curve. This trend in construction control was accepted on the basis that earth materials so placed would provide a fill of minimum permeability, minimum consolidation, minimum water content when saturated, and maximum ultimate stability. For practical reasons, limits for moisture control have generally been based upon moisture contents which would provide some fraction (as 98 per cent) of maximum laboratory density. The concept is extremely general and is illustrative of the tendency to generalize about the performance of a soil mass without giving full consideration to all of the factors involved in the placing and loading of the mass.

The stability of high rolled-earth dams in which impervious materials are used has been found to be greatly reduced by the development of pore fluid pressure in a soil mass placed at a relative high moisture content. Pore pressure (also termed excess hydrostatic pressure) develops in the mass as it consolidates from loading during construction, and a corresponding reduction in effective particle contact pressure is produced. Construction pore pressures have previously been estimated on the basis of rational formulas and laboratory tests,[2, 3] and have been observed extensively in earth dams by the use of field test installations.[4] The magnitude of the pore pressure is dependent upon the compressibility of the pore fluid (air and water), the volume change of the mass under load, and permeability of the mass and the surrounding material.[2, 3] Therefore, the effect of pore pressure on stability assumes increased importance as the placement moisture content is increased, as the fill loads become large, and as permeability of the materials used becomes small.

---

* Presented at the Meeting of Committee D-18 on Soils for Engineering Purposes held during the Fifty-first Annual Meeting of the Society, June 22, 1948, in Detroit, Mich.

[1] Head, Earth Materials Laboratory, U. S. Bureau of Reclamation, Denver, Colo.

It is well known that a soil mass which is placed at too low a moisture content will consolidate upon saturation, the amount of consodidation being dependent upon the placement moisture, the consolidation characteristics of the soil, and the final load conditions. Therefore, one important guide that laboratory tests and studies can furnish in rolled-fill construction is the establishment of suitable moisture control limits so that neither high pore pressure nor excessive consolidation after saturation will be produced during or after construction. Consolidation tests are used for determining these limits.

The information contained herein was determined in connection with the construction of four earth dams (Horsetooth, Soldier, Dixon, and Spring Canyon Dams) which form the Horsetooth Reservoir, Colorado-Big Thompson Project, Colo. Figure 1 shows the maximum section of the Spring Canyon Dam.

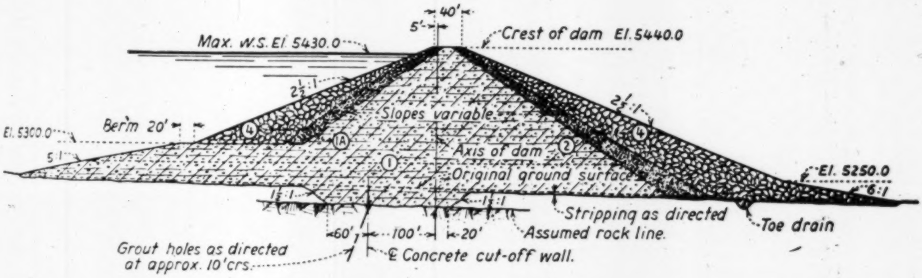


FIG. 1.—Spring Canyon Dam. (Maximum Section.)

(1) Impervious material of clay, sand, and gravel graduated in coarseness toward outer slopes, compacted in 6-in. layers.
(1-A) Semipervious sand and gravel compacted in 12-in. layers.
(2) Rock fines compacted in 12-in. layers.
(4) Rockfill graduated in coarseness toward outer slope.

LABORATORY TESTS

Selection of Samples:

Because of the large number of laboratory tests usually required to determine the placement moisture limits, samples representing the coarse, average, and fine materials in a borrow area are usually selected for the detailed consolidation tests required. This selection is made on the basis of classification and standard laboratory tests for gradation, compaction, and permeability. The soil selected for detailed discussion herein is identified as sample No. 3W-364 and is

---

[2] L. W. Hamilton, "The Effects of Internal Hydrostatic Pressure on the Shearing Strength of Soils," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1100 (1939).

[3] J. W. Hilf, "Estimating Construction Pore Pressure in Rolled Earth Dams," Paper No. 15.14, Second International Conference on Soil Mechanics and Foundation Engineering.

[4] W. W. Daehn and F. C. Walker, "Ten Years of Pore Pressure Measurements," Paper No. 15.18, Second International Conference on Soil Mechanics and Foundation Engineering.

representative of the "average" material from the borrow areas used in the construction of the Horsetooth Reservoir dams. The soil is classified as "sand with excess clay" (SF-clayey); the gradation is shown on Fig. 2. The density, consolidation, and pore pressure tests were conducted on the minus No. 4 fraction of the sample.

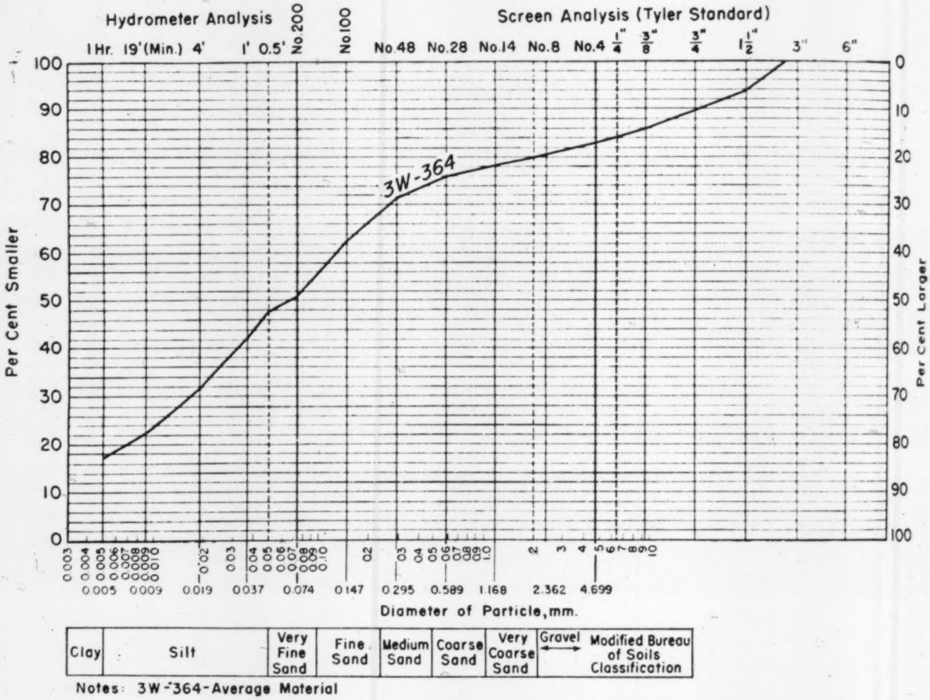


FIG. 2.—Placement Moisture Control. Laboratory Sample 3W-364. (Gradation Test)

Density Test:

Moisture-density (compaction) curves are determined for each material to be tested in a moisture limit program. The standard test generally used is similar to the A.S.T.M. Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T)[5] except that a $\frac{1}{20}$ cu. ft. compaction cylinder is used and the height of hammer drop is 18 in. The compaction test data used as the placement control for the consolidation tests on the Horsetooth soils were made on the basis of 33 blows per layer to simulate more nearly the compaction secured by the heavy rollers used on this project. The 33-blow compaction curve for the sample discussed herein is included in Fig. 3.

Consolidation Tests:

A fixed ring consolidometer is used for performing the consolidation tests. The equipment is described as follows. A platform scale is used for the loading equipment. The axial load is applied by means of a screw jack and yoke which apply the load through the specimen to the scale platform. The load is measured on the scale beam. The specimen container consists of a base plate and a brass ring of $4\frac{1}{4}$ in. inside diameter and

---

[5] 1946 Book of A.S.T.M. Standards, Part II, p. 1466.

$1\frac{1}{4}$ in. deep. The ring contains the soil specimen. Top and bottom porous disks allow complete drainage during consolidation. A strain gage, mounted to the base of the consolidometer and resting on the loading yoke, is used to measure the thickness change of the specimen during the test. A permeameter tube is provided to saturate the specimen from the bottom porous disk during the test. Figure 4 shows the consolidometer assembly. An extension collar is attached to the ring for preparing the specimens. Remolded soils, as used in this testing program, are compacted into the ring container to a depth of $1\frac{1}{2}$ in. at desired moisture and density conditions. The collar is then removed and the specimen is trimmed to a height of $1\frac{1}{4}$ in.

Moisture contents of approximately 2, 4, and 6 per cent below and 2 per cent above optimum moisture content and corresponding dry densities from the 33-blow compaction curve were selected for the placement conditions of the moisture limit tests discussed herein. Four specimens were prepared at each of these conditions, and the specimens were consolidated under single increment loadings of 25, 100, 175, and 250 psi. The con-

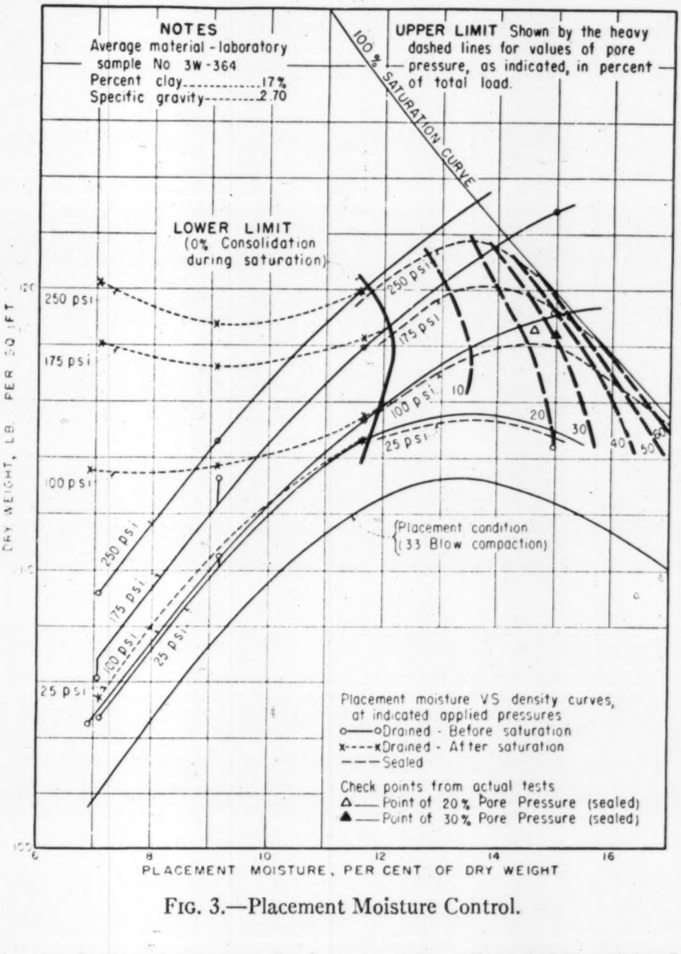


FIG. 3.—Placement Moisture Control.

solidated density was determined before and after saturation for each of the 16 specimens. The load increments were selected to cover the range of loadings anticipated for the soils used in the dam.

Additional specimens placed at other moisture contents within the same moisture range would be desirable, if time and facilities permit, to secure additional data for developing the limit curves.

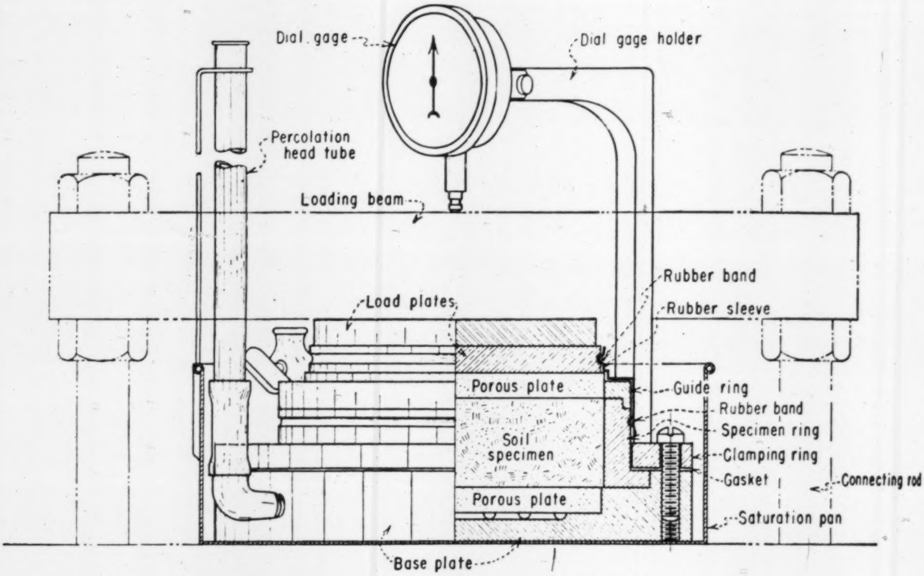

Dial gage
Dial gage holder
Percolation head tube
Loading beam
Rubber band
Load plates
Rubber sleeve
Porous plate
Guide ring
Rubber band
Soil specimen
Specimen ring
Clamping ring
Gasket
Connecting rod
Porous plate
Saturation pan
Base plate

FIG. 4.—One-Dimensional Consolidometer Specimen Container.

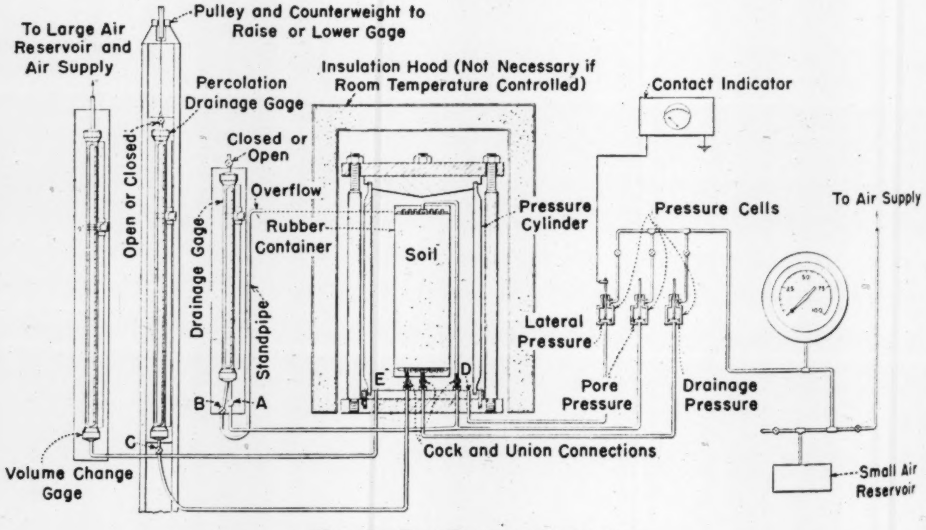

To Large Air Reservoir and Air Supply
Pulley and Counterweight to Raise or Lower Gage
Insulation Hood (Not Necessary if Room Temperature Controlled)
Contact Indicator
Percolation Drainage Gage
Open or Closed
Closed or Open
Overflow
Rubber Container
Soil
Pressure Cylinder
Pressure Cells
To Air Supply
Drainage Gage
Standpipe
Lateral Pressure
Pore Pressure
Drainage Pressure
25
50
75
100
E
D
B
A
C
Cock and Union Connections
Volume Change Gage
Small Air Reservoir

FIG. 5.—Pore Pressure Apparatus.

Pore Pressure - Consolidation Test:

The pore pressure - consolidation test is used to furnish data for the study of pore pressure and the related factors of permeability and consolidation. The pore pressure test equipment, as shown on Fig. 5, is described briefly as follows: a cylindrical soil specimen $3\frac{1}{4}$ in. in diameter and 3 in. high is encased in a thin-wall rubber sleeve which is clamped to perforated metal end plates, the end plates are connected to tubes that may be opened or closed to control drainage from the specimen and are also connected to no-flow pressure cells for pore pressure measurements. The specimen and container are placed in a pressure chamber and the sealed specimen is surrounded by water. The specimen is loaded by applying controlled air pressure to the water in the pressure chamber. A calibrated glass gage, located between the pressure chamber and air supply tank, is used to measure the volume of water flowing to or from the pressure chamber; this flow can be interpreted as volume change of the specimen. Remolded soil specimens are prepared in a split cylinder container at the desired moisture and density conditions by compacting in layers with a small compaction hammer.

Pore pressure tests were conducted in this testing program for the purpose of checking calculated pore pressure data. Three specimens were prepared at approximately 11, $13\frac{1}{2}$, and 15 per cent and at the corresponding densities obtained from the compaction curve. After preparation, the sealed specimens were placed in the consolidometer pressure chambers, and constant hydraulic pressures of 100 psi. were applied to all faces of the specimens. No drainage was allowed during the test, and the pore pressures were measured through the bottom end plate by the no-flow pressure cells. In this case it was desired to establish a relation between volume change and pore pressure for various placement conditions. The 100-psi. loading was selected only because it provided consolidation and pore pressure data within the desired ranges.

LOWER PLACEMENT MOISTURE LIMIT

The lower limit is defined as the lowest placement moisture content at which saturation will have no effect on consolidation. That is, the placement moisture must not be so dry that additional consolidation will take place when the soil mass becomes saturated under the fill load being considered. The data for determining the limiting moisture contents are presented in terms of dry density, placement moisture, and applied pressures.

As shown in Fig. 3, the four solid lines which were drawn through the unsaturated, consolidated dry densities obtained at each placement moisture condition provide the consolidated density *versus* placement moisture curves for each respective load condition before saturation. These pressure lines show the unsaturated, consolidated dry densities that will be obtained under the indicated applied pressures for various placement moistures when drainage is permitted. Similarly, the four dotted lines, which were drawn through the saturated consolidated dry densities obtained at each placement moisture condition, provide the consolidated density *versus* placement moisture curves after saturation without change in load. The difference between the solid and dotted lines is the effect of saturation on consolidation for the respective loads applied. The intersection of these two lines gives the placement moisture condition at which no additional consolidation will occur upon saturation. The heavy solid line plotted through these points of intersection therefore represents the lower limit of placement moisture for the respective fill pressures when consolidation due to saturation is to be avoided.

UPPER PLACEMENT MOISTURE LIMIT

The upper limit is defined as the highest placement moisture content which can be tolerated without producing serious construction pore pressures and thereby reduce the safety factor of the dam to an unacceptable amount. As practical limitations make it necessary to vary this limit within various parts of the structure, the upper limit lines are given for several pore pressure values in per cent of total load.

The upper limiting lines are calculated from the results of the one-dimensional consolidation tests by the following equations, by assuming that the specimens are completely sealed:

$$f = \bar{f} + u \dots\dots\dots (1)$$

where:

f = total stress (fill load),

$\bar{f}$ = effective stress (grain to grain pressure causing consolidation), and

u = pore pressure.

$$u_c = u_a\left(\frac{V_{ao} + hV_w}{V_{ac} + hV_w} - 1\right) \dots\dots (2)$$

or

$$u_c = \frac{u_a \Delta V}{V_{ao} + hV_w - \Delta V} \dots\dots\dots (3)$$

where:

u_c = pore pressure after consolidation (gage pressure),

u_a = initial pore pressure (usually considered as atmospheric pressure—absolute pressure),

V_{ao} = initial volume of free air in the soil mass (per cent of total initial volume),

V_{ac} = consolidated volume of free air in the soil mass (per cent of total initial volume),

ΔV = $V_{ao} - V_{ac}$ = volume change from consolidation (per cent of total initial volume),

V_w = volume of water in the soil mass (per cent of total initial volume), and

h = capacity of water to dissolve air from Henry's law (approx. 0.02).

Equation 1 shows a simple relation between total stress and effective stress, Eq. 2 was shown by L. W. Hamilton,[2] and Eq. 3 is similar to Eq. 2 but is expressed in a slightly different manner for easier use.[3] Equations 2 and 3 are based on Boyle's law for the compressibility of gases and Henry's law for the solubility of air in water at constant temperature. The assumption is made, in using these equations for computing pore pressures, that if a soil containing air, water, and solids is loaded without permitting drainage, it will consolidate, and the consolidation occurs as volume change in the air. The air will not only develop pressure according to Boyle's law, but it will dissolve in water according to Henry's law by a weight proportional to the pressure and to the volume of water present.

The one-dimensional consolidation test, as conducted for this study, allows complete drainage and provides consolidation data for effective loads of 25, 100, 175, and 250 psi. From this information, calculations were made to determine the total applied pressure (pore pressure plus effective pressure) necessary to produce the same consolidation that would occur if the soil were sealed from drainage. These consolidation data which were computed are shown for the various pressures by the light dotted lines on Fig. 3. The lines were obtained by interpolation for the total pressures desired from the computed data. The heavy dashed lines which cross these light dotted lines represent the pore pressures which will be obtained for the fill pressures and placement moistures indicated. The positions of these pore pressure lines were obtained by calculating the pore pressures at points along the total pressure lines (expressed in per cent of the total pressure) from Eq. 3.

As previously mentioned, pore pressure-consolidation tests were conducted to check the calculated pore pressure data. The placement moisture *versus* pore pressure and placement moisture *versus* consolidation curves for these tests are shown in Fig. 6. Two triangular points are plotted on Fig. 3 to show the relation of the pore pressure values obtained from the laboratory tests and the computed pore pressure curves. The consolidated density (sealed) and the placement moisture values for plotting these points were obtained from the curves shown in Fig. 6. From these curves it can be seen that 20 per cent pore pressure occurs at 14.6 per cent placement moisture for a consolidated density of 118.7 lb. per cu. ft., and 30 per cent pore pressure occurs at 15.0 per cent placement moisture for a consolidated density of 118.5 lb. per cu. ft. A reasonable check was obtained for the calculated pore pressure values and the values determined by the laboratory tests. By performing additional pore pressure-consolidation tests, a number of pore pressure-density-placement moisture values could be determined and interpreted as curves similar to the calculated pore pressure curves. Calculated data are usually used, however, because it is not practical to conduct the necessary number of pore pressure tests that would be required.

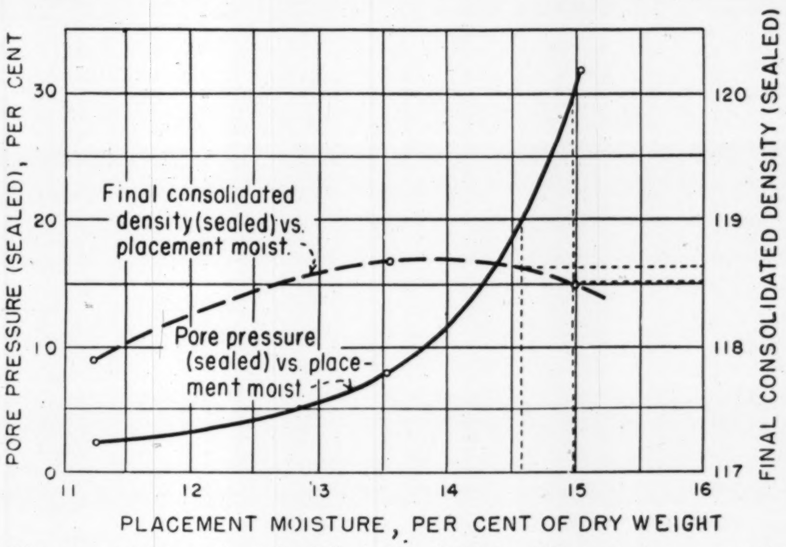


FIG. 6.—Placement Moisture Control. Sample No. 3W-364. Effect of placement moisture on pore pressure (sealed) at 100 psi. applied pressure.

USE OF MOISTURE LIMIT DATA

As an illustration of the use of the moisture limit curves, refer to Fig. 3. When placing this material in locations where it will be subjected to fill loads of 175 psi., the lower limit of placement moisture will be 12.1 per cent. The material, if placed above this moisture content, will be consolidated by the weight of the overlying embankment and will not consolidate an additional amount from saturation of the structure. For the same fill load, the upper limit for placement moisture will be 14.4 per cent for an allowable pore pressure of, say, 30 per cent, where pore pressure is expressed in per cent of total load.

Moisture limit data may be expressed in terms of the penetration resistance of standard Proctor needles, if desired. Such an expression of the moisture limit data is easily applied in field control procedures.

THE IMPORTANCE AND PRACTICAL USE OF RELATIVE DENSITY IN SOIL MECHANICS[*]

By Donald M. Burmister[1]

Relative density has been known in soil mechanics terminology for some time, but the practical importance and use of relative density as a fundamental concept in soil mechanics and as a powerful tool in the interpretation and correlation of soil behavior have not been fully realized. Relative density not only provides a common scientific basis for comparing the unit weight and degree of compactness of one soil with those of another soil of quite different character, but also brings the behavior characteristics of soils together on a common basis in consistent and practically useful relationships.

It is the purpose of this paper to focus attention on the possibilities and practical uses of relative density in soil engineering work. Relative density relationships not only give a clearer insight into and a better understanding of soil behavior, as influenced by the individual characteristics of soils, but also permit a more complete statement to be made of the physical laws governing soil behavior in graphical forms with relative density as the common argument. This may be more useful than attempts to give rigorous mathematical treatments of such complex behavior characteristics as compressibility, angle of friction, permeability, relative supporting value, etc. The examples of the relative density relationships discussed herein, although not fully developed at the present time, serve to illustrate the importance and possibilities of relative density in soil engineering work.

Definition of Relative Density

Relative density, a more expressive and simpler term than degree of density or degree of compaction, is defined[2,3] on a voids ratio basis as follows:

Relative density, D_R, in per cent

$$= \left(\frac{e_L - e_N}{e_L - e_D}\right) 100 \ldots (1)$$

where e is the voids ratio of the soil and the subscript, L refers to soil in the loosest state or minimum density, D refers to soil in the densest state or maximum density, and N refers to the soil in the natural or artificially compacted state under consideration.

Relative density may also be expressed in other convenient and useful forms, based upon Eq. 2 and 3, as follows:

Saturated moisture content,

$$w', \text{ in per cent} = (e/G_s)100 \ldots (2)$$

Unit dry weight, lb. per cu. ft.,

$$W_s = \frac{62.4G_s}{1 + e} \ldots (3)$$

where G_s is the specific gravity of the soil grains.

---

[*] Presented at the Meeting of Committee D-18 on Soils for Engineering Purposes held during the Fifty-first Annual Meeting of the Society, June 22, 1948, in Detroit, Mich.

[1] Associate Professor of Civil Engineering, Columbia University, New York, N. Y.

[2] Tentaive Definition of Terms and Symbols Relating To Soil Mechanics (D 653 - 42 T), 1946 Book of A.S.T.M. Standards, Part II, p. 1473.

[3] "Soil Mechanics Nomenclature," A.S.C.E. Manuals of Engineering Practice, No. 22 (1941).

Relative density, saturated moisture content basis,

$$D_R = \left[\frac{w'_L - w'_N}{w'_L - w'_D}\right] 100 \ldots\ldots (4)$$

Relative density, unit dry weight basis,

$$D_R = \left[\frac{\dfrac{1}{W_L} - \dfrac{1}{W_N}}{\dfrac{1}{W_L} - \dfrac{1}{W_D}}\right] 100 \ldots\ldots (5)$$

The relative density diagram given in Fig. 1 can be used conveniently for determining relative densities and unit dry weights, provided that the minimum and maximum densities are known, as illustrated.

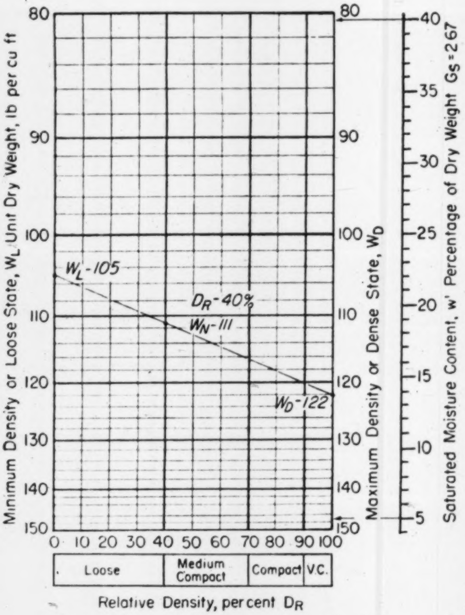


FIG. 1.—Relative Density Diagram.

MAXIMUM AND MINIMUM DENSITIES

It is important to note that relative density must be defined on the basis of the minimum and maximum densities as reference for the 0 per cent and 100 per cent relative densities, respectively. The minimum and maximum densities are as significant characteristics of granular soils as the liquid and plastic limits are for clay-soils. These references are defined from two points of view, both of which are important: namely,

(*a*) soils in the natural state as formed by the processes of weathering or as deposited by such geological processes as sedimentation in flowing and quiet water, wave action, wind action, and glacial action; and (*b*) soils artificially placed and compacted in embankments by the common methods of construction.

The maximum and minimum densities, as references, for granular soils in the natural state with less than about 35 per cent of silt (material passing the No. 200 sieve of a non-plastic character) can be conveniently and reliably determined on the soil in the dry condition. The maximum density is obtained by compacting the soil in ½-in. layers in a cylinder of known volume with a light tamping action of a "Vibro-tool" (Burgess Battery Co.) fitted with a tamping foot about 1½ in. in diameter. The end point can be readily judged for each layer by the fact that the surface of the layer tightens up and shows no appreciable movement when very lightly tamped at the end of the operation. This maximum density reference is a significant characteristic of a given soil and represents practically the maximum density attainable in the laboratory. Experience shows that this maximum density has seldom been exceeded in natural deposits of granular soils.

The minimum density is obtained by spreading the soil in thin uniform layers about ½-in. thick from a funnel fitted with a special large spout at least 1 in. in diameter. This minimum density reference is a practical physical limit to the loosest state in which a soil can be placed in the dry condition. Experience shows that natural deposits of granular soils have seldom been found in a more loose condition.

The liquid and plastic limits for clay-soils[4] in the natural state, having a medium plasticity or higher (plasticity index greater than 10) provide convenient 0 per cent and 100 per cent references, respectively, for relative density determinations in Eq. 4. The liquid limit moisture content represents a characteristic minimum density, which is typical of many soft natural clay and organic silt deposits. The liquid limit as the 0 per cent relative-density reference refers to the natural moisture content only and does not refer to the natural undisturbed consistency or shearing strength. The plastic limit moisture content represents a characteristic maximum density which is seldom exceeded in natural deposits except where subjected to very heavy overburden pressures or to drying out processes by evaporation of water in exposed surface deposits.

The maximum density for silty and clayey soils to be compacted in embankments, should be determined by the standard compaction test, A.S.T.M. Tentative Method D 698 - 42 T,[5] or by the modified A.A.S.H.O. compaction test.[6] These, however, are temporary values, which may be increased somewhat as better and more effective methods are developed for compacting soils.

The minimum density, which is important as the 0 per cent relative density reference, is influenced by more or less important bulking effects. A common loose field condition is obtained by dumping moist soil from a truck into a cubic foot container. In the laboratory a minimum density should be determined in parallel with the standard compaction test as a convenient and practical physical limit. At each increment of moisture, the moist soil is placed in the cylinder 1-in. layers and lightly compacted by one coverage of 5 applications of the standard $5\frac{1}{2}$ lb. rammer applied gently as a static loading.

TABLE I.—RELATIONS BETWEEN RELATIVE DENSITY AND RELATIVE SUPPORTING VALUE OF NATURAL SOIL DEPOSITS AND OF ARTIFICIALLY COMPACTED SOILS.

(a) GRANULAR NON-PLASTIC SOILS. COMPACTNESS.

Relative Density, per cent........−20	0	40	70	90 100
Compactness....................Bulking	Loose	Medium	Compact	Very compact
Relative Supporting Values:				
Silt	Poor to Fair	Fair to Good	Good—very good	Excellent
Sand	Fair	Good	Very good	Excellent
Gravel	Fair to Good	Good to very good	Very good plus	Excellent

(b) PLASTIC CLAY-SOILS. CONSISTENCY OF ARTIFICIALLY COMPACTED CLAY-SOILS OF LOW PLASTICITY AND HIGHER

Relative Density, per cent........−20	0	40	70 85	90 100
Consistency	Very soft	Soft	Med. Soft	Stiff
Relatively Supporting Value..........	Practically none	Very poor	Poor—fair	Fair—good

---

[4] Clay-Soil is defined as material passing the No. 200 sieve, which exhibits plastic properties and clay qualities within a certain range of moisture content and has considerable strength when air dried. See Report of Committee VII on Foundations and Soil Mechanics on "Definitions of Soil Components," Civil Engineering Div., Am. Soc. Engineering Education, *Civil Engineering Bulletin*, Vol. 12, No. 2, November, 1947.

[5] Tentative Method of Test for Moisture-Density Relations of Soils, 1946 Book of A.S.T.M. Standards, Part II, p. 1466.

[6] Method of Test for the Compaction and Density of Soil, Standard Specifications for Highway Materials and Methods of Sampling and Testing, Am. Assn. State Highway Officials, Part II, p. 212 (1947).

SIGNIFICANCE OF RELATIVE DENSITY

Experience has shown conclusively that the compactness of natural deposits of soil and of soils compacted in subgrades of highways and of airport runways and in embankments has a most important influence on the supporting value of the soil. The concept of relative density focuses attention, first of all, on the important fact that relative density is a significant state of the grain structure of granular soils which controls relative supporting values. Relationships defining relative supporting values of granular soils in terms of relative density are given tentatively in Table I (*a*) with the characteristic regions defined on the basis of the author's experience.

Studies have shown that various geological processes tend to form soil deposits in characteristic states of compactness. Practically all natural deposits of sand and of silt formed by the processes of sedimentation in flowing or quiet water, or by wind action, are in a loose state and have relative densities less than 40 per cent. The coarser sands deposited by flowing water are more likely to have relative densities between 20 and 40 per cent. The finer the soil, the lower will be the relative density when deposited by flowing water, in quiet water, or by wind action, and generally less than 20 per cent. It will seldom be less than 0 per cent (or minus). Gravelly soils of glacial outwash or piedmont origin are more likely to be deposited in the lower range of the medium compact state with relative densities slightly greater than 40 per cent. Beach deposits of medium and fine sands formed by wave action are likely to be in quite a dense state with relative densities greater than 70 per cent.

The characteristic states of compactness in which geological processes tend to form soil deposits will have a determining influence on their relative supporting values. There is an important distinction, however, between relative supporting value and bearing value. Relative supporting values can be conveniently expressed in general terms with regions defined on the basis of relative density, which describes the significant state of the grain structure of granular soils, as tentatively defined in Table I (*a*). Bearing value, on the other hand, is expressed in numerical values for the practical purposes of the design of foundations. First of all it is essential to recognize that bearing value is not an inherent single-valued soil property. It must be evaluated in each case to apply satisfactorily to the specific set of conditions that control in a given situation. These conditions include: (1) the permissible total and differential settlements, which will not impair the usefulness of a structure, or will not cause damage that will affect the integrity and safety of a structure; (2) the type and size of structure, character of loading, and size and arrangement of footings or the type of foundation; and (3) the depth below the surface of the ground, the subsurface soil conditions, etc. For practical purposes of design, relative supporting values must be properly evaluated into terms of numerical bearing values on the basis of experience and of foundation investigations, which adequately take into account the controlling conditions in a given situation.

This concept is implied in the tentative designations of relative supporting values for granular soils given in Table I (*a*). Natural deposits and fills of granular soils in the loose state have poor relative supporting values because the settlements are likely to be objectionable even under light loads. Soils in the loose state should be considered to have no bearing value, unless load tests or experience demonstrate the safe load limits in terms of permissible settlements, especially when subjected to vibrational loadings. Deposits of soil in the medium compact state have fair to good relative supporting values, because the safe load limits are higher. They would provide adequate support for highway pavements laid directly on such subgrades. On the other hand such subgrades would not provide satisfactory permanent support for airport runway pavements under heavy wheel loads and vibrational effects because of the objectionable consolidation and consequent roughening and cracking of pavements. A minimum relative density of 90 per cent is required in order to limit objectionable consolidation and settlement under heavy vibrational loadings.

It is to be noted that recognition is given in Table I (*a*) to the important fact that the relative supporting value is dependent not only on the relative density of the soil but also on the coarseness of the soil. For the same relative density, the coarser soils, and particularly the gravels, are less compressible because of the greater resistance of the large particles to displacement. Hence in terms of permissible settlements such soils have higher relative supporing values, as noted.

In natural deposits of granular soils, the grain structure of the soil is due only to the arrangement of the soil grains consistent with the state of compactness, which can be adequately expressed in terms of relative density. Hence relative density is a satisfactory index of relative supporting value, giving due consideration to the coarseness of the soil. On the other hand, in natural deposits of residual clay-soils and of sedimentary clay-soils the natural structure and the state of aggregation of the soil particles dominate and give a strength and a supporting value to such soils considerably greater than that to be expected on the basis of relative density alone. For compacted clay-soils it is the consistency or the resistance to plastic displacements of the compacted soil after it has reached a state of capillary saturation in equilibrium with its environment which determines the relative supporting value that can be permanently relied upon. A clay-soil compacted considerably on the dry side of the maximum density may appear to be very hard and firm when first compacted, but with capillary saturation will become very soft with a greatly reduced supporting value.

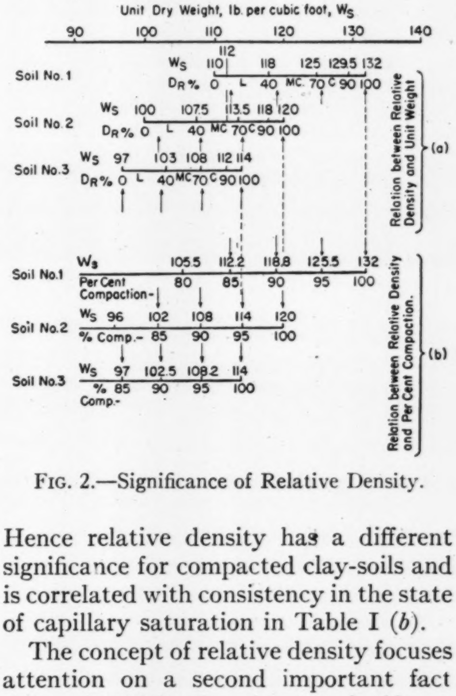

Unit Dry Weight, lb. per cubic foot, W_S
90
100
110
120
130
140
Soil No. 1
W_S 110 112 118 125 129.5 132
D_R% 0 L 40 MC. 70 C 90 100
Soil No. 2
W_S 100 107.5 113.5 118 120
D_R% 0 L 40 MC 70 C 90 100
Soil No. 3
W_S 97 103 108 112 114
D_R% 0 L 40 MC 70 C 90 100
Relation between Relative Density and Unit Weight
(a)
Soil No. 1
W_s 105.5 112.2 118.8 125.5 132
Per Cent Compaction - 80 85 90 95 100
Soil No. 2
W_S 96 102 108 114 120
% Comp. - 85 90 95 100
Soil No. 3
W_S 97 102.5 108.2 114
% Comp. - 85 90 95 100
Relation between Relative Density and Per Cent Compaction.
(b)

FIG. 2.—Significance of Relative Density.

Hence relative density has a different significance for compacted clay-soils and is correlated with consistency in the state of capillary saturation in Table I (*b*).

The concept of relative density focuses attention on a second important fact that unit weight alone is not a significant index of relative supporting value, either in natural deposits of soil or in artificially compacted soils, which is illustrated in Fig. 2 (*a*). For each soil there are characteristic maximum and minimum densities representing for practical purposes definite physical limits. In Fig. 2 (*a*) three granular soils may, for example, have exactly the same unit dry weight of 112 lb. per cu. ft. in the natural state, or in a compacted fill, but they will have radically different relative supporting values, as defined in Table I (*a*) on the basis of relative density. Soil No. 1 will have only a poor relative supporting value because it is in about its loosest possible state; soil No. 2 will have a fair to good relative supporting value, because it is in a medium compact state; while soil No. 3 will have an excellent relative supporting value, because it is in a very compact state. Thus relative density is a significant state of the grain structure of a granular soil that controls in a given situation. But it must also be recognized that potentially soil No. 1 would be the best of the three soils if it were compacted to its maximum density, and soil No. 3 would then be the poorest.

The concept of relative density focuses attention on a third important fact, namely, that the commonly used term, "per cent compaction", expressed as a simple percentage of the maximum density only, is not significant but is actually misleading, because it is based upon the obvious fallacy that zero per cent compaction can represent a unit weight of zero for any soil, whereas there is always a definite physical limit to the minimum density or loosest state. This important fact is illustrated in Fig. 2 (*b*) for the three soils previously discussed. A field control requirement of a certain per cent compaction gives a false sense of a good degree of compaction, which does not in fact exist. To the average engineer or contractor 100 per cent compaction represents perfection and 90 per cent compaction, according to common standards of engineering practice, is considered to be very good indeed. But a study of Figs. 2 (*a*) and 2 (*b*) reveals the following state of affairs, which is typical of all soils: first, 90 per cent compaction is only slightly above 40 per cent relative density, or barely in the medium compact state with only a fair relative supporting value for granular soils and with a very poor relative supporting value for clay-soils after capillary saturation; and second, 95 per cent compaction, which is usually considered to be practically perfection, is in the lower compact state, with only a fair relative supporting value for clay-soils after capillary saturation.

It is evident that field compaction requirements and specifications based on percentage compaction are not good enough for the construction and treatment of subgrades and of high quality base courses for airport runway pavements intended to support heavy airplane wheel loadings, particularly under the vibrational effects. Such requirements are inadequate because they cannot properly fix the lower density limit considered permissible and acceptable in earthwork construction, which will minimize objectionable consolidation of granular soils and objectionable lateral plastic displacements of clay-soils under such loadings. Only 100 per cent compaction has any real practical significance. The use of term "per cent compaction" in specifications should therefore be discontinued.

A relative density basis for specifying the requirements for compaction is adequate and provides a satisfactory index of the real state of perfection of compaction. On the basis of the minimum and maximum densities for each class of soils, the lower density limit can be properly fixed from experience on a relative density basis, which would be considered permissible and acceptable for the given earthwork construction. The minimum permissible relative density should be sufficiently high for granular types of subgrades and base courses to keep consolidation within non-objectionable values, and for clay-soil types of subgrades to keep plastic displacements, softening, and loss of support with capillary saturation within non-objectionable values, so as to insure pavement smoothness and integrity for the intended life of the pavement. Admittedly a specification requiring a minimum of 70 per cent relative density for the compaction of all fills for airport runways, and a minimum of 90 per cent relative density for possibly the upper 2 to 3 ft. of all subgrades in cuts and in fills and for all base courses is high, but it is necessary in order to insure high permanent supporting values required for runway pavements.

RELATIVE DENSITY RELATIONSHIPS OF SOIL BEHAVIOR

Relative density, as a common argument and as a significant state of the grain structure of granular soils, plays an important rôle in soil mechanics in bringing complex phenomena of soil behavior together on a common basis and in establishing basic physical relationships that control soil behavior. The inherent characteristics of the granular soil material that shape behavior are grain size distribution and dominating grain shapes. Some of the basic characteristics of the grain structure of granular soils that control behavior are relative density, initial stress conditions, degree of confinement, percentage saturation and pore pressures. A number of relative density relationships, although not completely developed at the present time, are presented in order to illustrate the possibilities of developing fundamental physical relationships and laws of behavior of granular soils with regard to relative density and to grain size distribution as the principal characteristics.

Penetration Test Relationships:

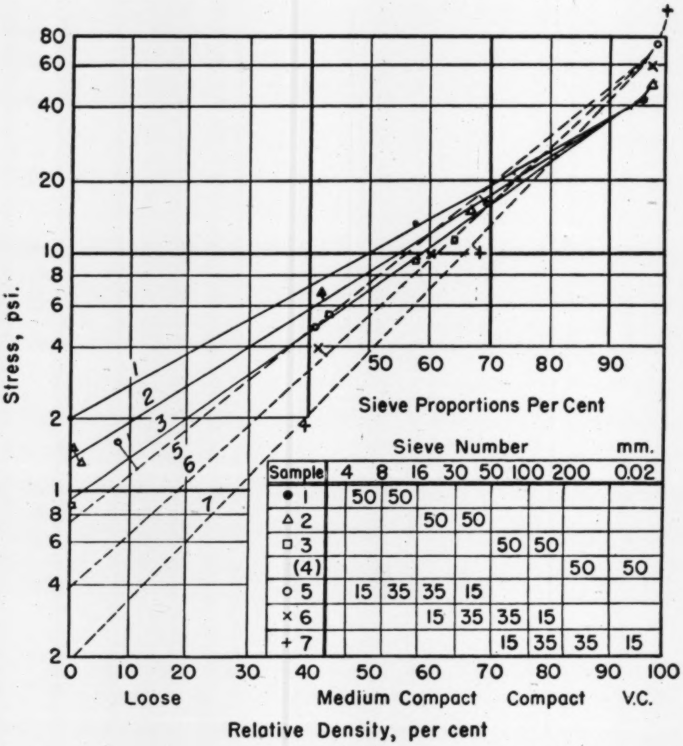
The best known penetration test is the California Bearing Ratio test.[7] The California Bearing Ratio has been used extensively as an index of the relative supporting value of compacted soils. However the behavior characteristics of granular soils and of clay-soils differ in such important respects that the same criteria are not directly applicable to both. The strength properties of granular soils are markedly affected by such characteristics of the grain structure as the degree of confinement and the initial stress conditions imposed on the soil as, for example, by a surcharge loading and confinement such as produced by a pavement on a subgrade or by placing a footing below the surface of the immediately adjacent ground. The strength properties of clay-soils, on the other hand, under fairly rapid loading conditions may not be affected at all by these physical factors or only to a limited extent, but are markedly affected by the resistance to plastic displacement that can be mobilized within the clay-soil mass.

Research on the California Bearing Ratio test by Tanner[8] on a series of granular soils has disclosed a number of important facts. First, the standard penetration of 0.1 in. for the CBR value was not found to be significant for granular soils, because for relative densities greater than about 70 per cent with and without a surcharge there is a peak of penetration resistance, which is followed thereafter by a characteristic and pronounced stress drop-off. A value of 0.01 in. penetration was therefore selected, as being more significant, which falls on the linear portion of the stress-penetration curve below the peak of penetration resistance for all types of granular soils tested. A preliminary light seating load was required in order to obtain consistent results. Second, definite relationships were found to exist between relative density and the stress at a penetration of 0.01 in. under a surcharge, as shown in Fig. 3, in which there is a logarithmic increase in stress with increase in relative density. Third, the relationships in Fig. 3 show that grain size distribution plays in important rôle in shaping soils behavior. At low relative densities, the stress is greatly influenced by the coarseness of the soil and by the range of grain sizes, being higher for coarse sands than for fine sands and for a narrow range of grain sizes than for a wide range of grain sizes, due to the appreciably greater resistance to displacement of the individual grains. In the dense state the stress becomes independent of grain size distribution. These facts substantiate the relationships given tentatively in Table I (*a*).

---

[7] O. J. Porter, "The Preparation of Subgrades," *Proceedings*, Highway Research Board, Part II, p. 324 (1938).

[8] D. N. Tanner, "Investigation of the California Bearing Ratio Properties of Sands," Thesis No. 555 for the M. S. Degree, Department of Civil Engineering, Columbia University, New York, N. Y., June, 1948. (Not published.)



Sample		4	8	16	30	50	100	200	0.02
● 1		50	50						
△ 2				50	50				
□ 3						50	50		
(4)								50	50
○ 5		15	35	35	15				
× 6				15	35	35	15		
+ 7						15	35	35	15

FIG. 3.—Relations Between Resistance to Penetration and Relative Density.

Sand compacted in 4-in. diameter cylinder.
Stress on 1⅜-in. diameter plunger at 0.01 in. penetration.
Surcharge—2 psi.
After Tanner. Thesis No. 555 for M. S. Degree, 1948.
Department of Civil Engineering, Columbia University.

Driving Resistance Relationships of Sampler:

Approximate information as to relative supporting values of soils for foundations on natural soil deposits is obtained during sampling operations from the driving resistance of the sampler, expressed in blows per foot, or preferably blows per 6 in. Engineers have commonly used rough rule-of-thumb methods based on the blows to drive the sampler, but experiences have varied widely. It is important first of all to recognize that for granular soils the driving resistance of the sampler is an index of compactness, whereas for clay-soils it is an index of consistency or resistance to plastic displacement, which are two quite different phenomena. A more careful control of these procedures is required in order to obtain reasonably consistent practical information on the relative supporting values of soils from a field procedure in which there are so many variable elements.

To illustrate the possibilities of developing useful and practical relationships, data are presented in Fig. 4, in which tentative relative supporting values are designated in terms of the driving resistance of the sampler and of relative density. It is essential to recognize, as discussed in connection with Table I, that relative supporting values can be conveniently expressed in general terms. These however, must be properly evaluated for the purposes of foundation design into numerical bearing values to apply satisfactorily to the specific set of conditions that control in each given situation. The blows per foot required to drive a 3-in. M & P sampler under a driving energy of 5000 in-lb. are plotted against the corresponding relative densities of the samples of granular soils. The envelop curves represent about the limits to be expected between silts and gravels. The character of the soil should be known in order properly to evaluate the driving resistance of the sampler. The resistance to driving is greater in gravelly soils for the same relative density because of the greater resistance to displacement of the larger particles. Higher relative supporting values have been assigned to the coarser soils by designating tentative zones of relative supporting values in Fig. 4 which cut across the vertical relative density zones in the direction of increasing coarseness of the soil. This is in agreement with the relative supporting values tentatively assigned in Table I (*a*).

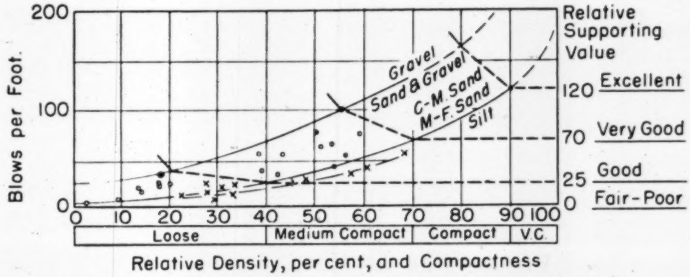


FIG. 4.—Relations Between Driving Resistance of Sampler in Blows per Foot and Relative Density for Granular Soils.

3-in. M. & P. Sampler.
Driving energy, (W × H) = 5000 in-lb.

In interpreting such data it must be recognized that the relative densities of the soils in the sample tube may be somewhat different from the actual relative densities of the undisturbed soil due to the sampling operation. For granular soil deposits in the loose state, the relative density of the soil in the sampler will be slightly too high as a result of the vibrating and compacting effect of driving the sampler. On the other hand, for granular soil deposits in the dense state the action of shearing through the soil and of displacing the soil particles will tend to loosen up the soil somewhat, and thus tend to make the relative density of the sample too low. These conditions will vary with the type and coarseness of the soil, the size and type of sampler, and with the driving energy expressed by the product of the weight of hammer in pounds and the height of drop in inches.

It may, however, be stated that the relative density obtained in the sampler, in general, will be consistent with the number of blows required to drive the sampler 1 ft. (or 6 in.) for a given driving energy and hence must also bear some consistent relationship to the natural relative density, since relative density together with coarseness is the principal physical factor mobilizing the resistance to driving of the sampler. In order to better interpret and evaluate the driving resistance relationships, the blows for each 6 in. should be recorded from the beginning of the sampling operation. Some evidence indicates that the relative density of the sample will not be significantly different from the actual values in the undisturbed soil, provided that the following conditions are reasonably satisfied: (1) that a thin wall Shelby tube is used; (2) that the length of the sample obtained is not more than four to six times the diameter of the sampler; and (3) that the blows required to drive the last 6 in. are not materially greater than for the preceding 6 in.

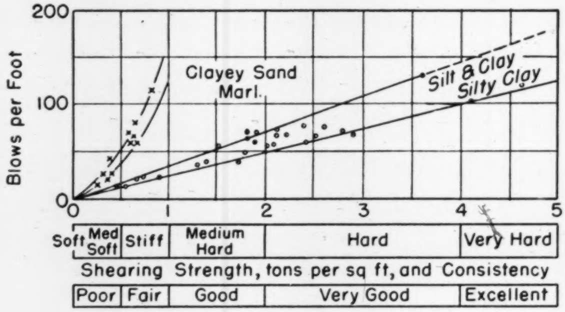


FIG. 5.—Relation Between Driving Resistance of Sampler in Blows per Foot and Shearing Strength for Clay-Soils.

3-in. M. & P. Sampler.
Driving energy, (W × H) = 5000 in-lb.
Unconfined compression tests on undisturbed samples.

The driving resistance relationships for clay-soils are also given in order to illustrate the differences in the phenomena and in order to provide a basis for judgment. The natural structure and the natural shearing strength in clay-soils in the undisturbed state are of greater importance than relative density in determining the relative supporting value of the soil. Correlations of the driving resistance in clay-soils with the shearing strength obtained by unconfined compression tests on undisturbed samples of good quality (least disturbed), (Fig. 5), illustrate the possibilities of developing useful and practical relationships. The relative supporting values are given directly in terms of shearing strength. However, it must be emphasized that before such relationships should be used, the "goodness" of the samples as to undisturbed quality must be properly evaluated and known. Samples of clay-soils of poor quality caused either by (*1*) improper methods of advancing and cleaning the casing and of obtaining the samples, (*2*) the disturbance caused by stones, shells, or other natural obstructions, or (*3*) interbedded layers, can give a completely false picture of a situation with regard to the relative supporting value of such soils and of the difficulties to be encountered during construction.

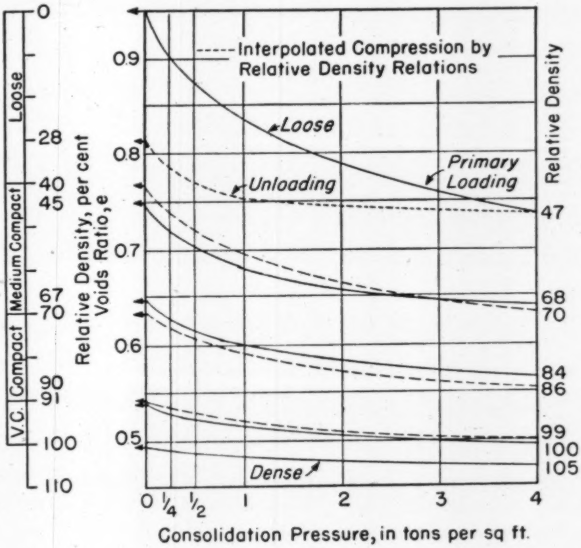


FIG. 6.—Pressure-Voids Ratio Curves and Relative Density Relations.
Soil used: "Coarse to fine sand, little silt."

Correlations of the driving resistance in the same and in similar soil conditions with different weights of hammers and heights of drop and with different sizes and types of samplers indicate that the following approximate relationships may be used tentatively for computing a new scale of values to be used in Figs. 4 and 5. These are based upon the driving energy used and upon the displacement area of the sampler.

New scale of blows per foot (or per 6 in.):

$$B^1 = B\,\frac{5000}{WH}\left[\frac{D_0^2 - D_i^2}{3.625^2 - 2.93^2}\right] \ldots .(6)$$

where B is the blows per foot in Figs. 4 and 5 for a 3 in. M & P sampler; W is the weight of the new hammer in pounds; H is the new height of drop in inches; and D_0 and D_i are the outside and inside diameters of the new sampler in inches.

Consolidation Relationships of Granular Soils:

The settlement of structures is due principally to the consolidation of the supporting soils under the stresses imposed by the foundation loads. The principal problem in the design of the foundations is to limit the total and the differential settlements to non-objectionable values, which will not impair the usefulness of the structure and will not affect the safety and the integrity of the structure. Although granular soils, in general, provide a satisfactory foundation support, the settlements of structures founded on deposits of loose granular soils may reach objectionable magnitudes under moderate to heavy foundation loads, particularly under vibrational loadings, where differential settlements become the controlling factor.

The compression curves of granular soils show a characteristic pattern with relative density and express the physical relationships governing compressibility in graphical form. This is illustrated in Fig. 6 for a typical soil identified as "Coarse to fine Sand, little Silt." For each soil there are characteristic maximum and minimum densities, determined by grain size distribution and by dominating grain shapes, which place the compression curves in a certain range of voids ratio. At low relative densities the soil is very compressible, particularly under light pressures. At 100 per cent relative density the soil becomes almost incompressible with a very flat compression curve. The full line compression curves of the series in Fig. 6 are test curves with the initial voids ratios and relative densities noted. The dotted compression curves were interpolated between the 0 and 100 per cent relative density compression curves corresponding to initial 40, 70, and 90 per cent relative densities. It is evident from the general agreement in form of the actual and the interpolated compression curves that there is a characteristic change in compressibility with initial relative density.

As a general law of compressibility of granular soils there is not only a characteristic pattern of compression curves for each soil with change in relative density from 0 to 100 per cent, but also a characteristic pattern of compression curves with change in initial voids ratio for different soils at 0 per cent and at 100 per cent relative densities, respectively. The respective curves are almost identical in form at corresponding initial voids ratios. Soils having high initial voids ratios are more compressible than soils having low initial voids ratios at the same relative density. The compressibility of different granular soils at 0 per cent relative density is very sensitive to grain size distribution and dominating grain shapes, which parallels their characteristic minimum densities. The finer-grained granular soils having high initial voids ratios at 0 per cent relative density are more compressible than the coarser grained soils having low initial voids ratios at 0 per cent relative density. On the other hand, all granular soils at 100 per cent relative density are relatively incompressible, the compressibility being almost independent of grain size distribution and of initial voids ratio.

If these relationships can be shown to hold generally for all types of granular soils, then the consolidation characteristics and the compressibility of granular soils for the initial loading conditions can be fully determined for the entire range of relative density. This is dependent on the compression curves for 0 per cent and for 100 per cent relative densities, respectively, being carefully determined for a series of granular soils covering the range of typical grain size distributions by suitable steps. A compression curve for a given grain size distribution corresponding to a certain relative density in the natural state or in the compacted state could then be readily interpolated for the practical purposes of estimating the settlement of structures or the shrinkage of fills under their own weight.

Angle of Friction Relationships for Granular Soils:

The determination of the angle of friction of granular soils is an important matter for the design of retaining structures and for the analysis of the stability of slopes in such materials. It is essential to know the probable range of the angle of friction for the field densities in which a backfill of a retaining wall or of a sheet-pile bulkhead may be placed or in which an embankment may be constructed. Figure 7 shows that characteristic relationships of angle of friction with relative density exist, which are reasonably consistent with respect to the grain-size distribution for the given soils. These angle-of-friction—relative-density relationships represent the failure conditions or maximum value of the angle of friction corresponding to the maximum or peak value of the shearing stress-strain curve for normal pressures up to 2 tons per square foot. For a narrow range of grain sizes, such as for Ottawa sand, the relationship is linear. When the soil is composed of a wide range of grain sizes, the relationship is linear to about 70 per cent relative density. Thereafter, there is a tendency for a characteristic curvature upward toward 100 per cent relative density due probably to the more marked interlocking, wedging, and bedding of the grains in the dense state. In the loose state at low relative densities, the angle of friction is quite sensitive to differences in the grain-size distribution of soils with a considerable spread in the values,

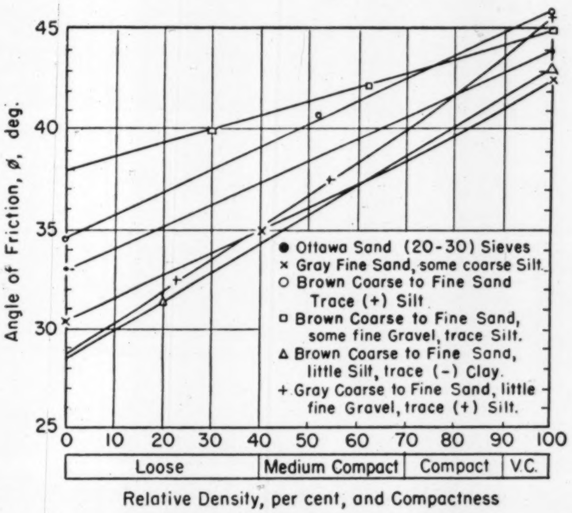


FIG. 7.—Angle of Friction—Relative Density Relations for Granular Soils.

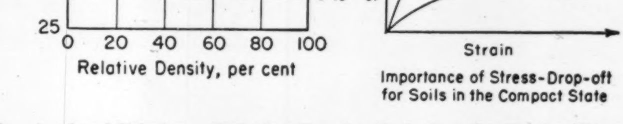


FIG. 8.—Angle of Friction—Relative Density Relations for Design Purposes.

whereas in the dense state the spread in the angle of friction is very much less. In general these relationships indicate that the coarse soils have somewhat higher angles of friction at all relative densities due to the greater resistance to displacement of the larger grains.

It is evident that, if shearing tests are made at 0 per cent and at about 70 per cent relative densities, reliable estimates of the probable range of the angle of friction can be made for design purposes that will bracket the limiting values for the probable range of field densities. In selecting values of the angle of friction to be used for design purposes, where soils have relative densities above the loose state, it is essential to take into account the important and characteristic shearing strength drop-off that occurs after the peak of strength, as illustrated in the shearing stress-strain relationships in Fig. 8. In the loose state, the shearing stress increases gradually to a maximum value at a large value of strain and remains constant thereafter with no stress drop-off. As the relative density increases, however, the shearing stress increases more rapidly to a definite peak of strength at considerably smaller strain values, and the shearing strength drops off in a characteristic fashion thereafter to an ultimate shearing strength somewhat higher than that in the loose state. The higher the relative density, the more important and pronounced does the strength drop-off become and the smaller is the strain at which the peak or maximum strength occurs. This characteristic strength drop-off is due to the expansion and decrease in relative density of compacted granular soils, which is always associated with shearing action in the denser states. Because of this strength drop-off, it is not possible to realize fully the important increase in the angle of friction obtainable by increased compaction of backfills or of embankments. It is, therefore, necessary to use a value of the angle of friction for design purposes somewhat less than that corresponding to the peak or maximum shearing strength. This is necessary because of the tendency for progressive failure conditions to develop in a backfill or in an embankment, due to differences in the strain conditions along different portions of the sliding curve of failure.

The practical range of values of the angle of friction for design purposes is shown tentatively in Fig. 8. Where somewhat unfavorable conditions tend to control a situation, which cannot be fully evaluated on a numerical basis, it is believed that the use of at least 50 per cent of the increase of the angle of friction (peak value) above that for the loose state is justified for design purposes, judging from the ultimate shearing strength obtained in the usual types of shearing tests. Where favorable conditions tend to control a situation, it is believed that the use of 75 per cent of the increase in the angle of friction above that for the loose state is justified for design purposes. It should not be higher than this value in any case because of the possibility of progressive failure conditions developing. These relationships are shown in Fig. 8. Thus reliable working values of the angle of friction can be obtained for design purposes that will bracket the limiting values for the probable range of field densities. It is important that the probable range of field densities be reliably determined in all cases where an angle of friction greater than that for the loose state is to be used for design purposes in order to be on the safe side and at the same time to realize some of the advantages of a higher degree of compaction of backfills and of embankments.

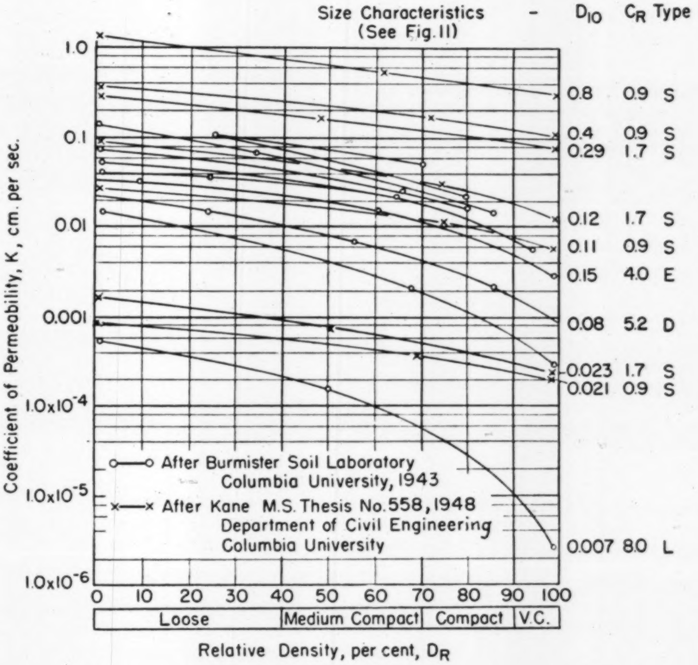


FIG. 9.—Permeability-Relative Density Relationships.

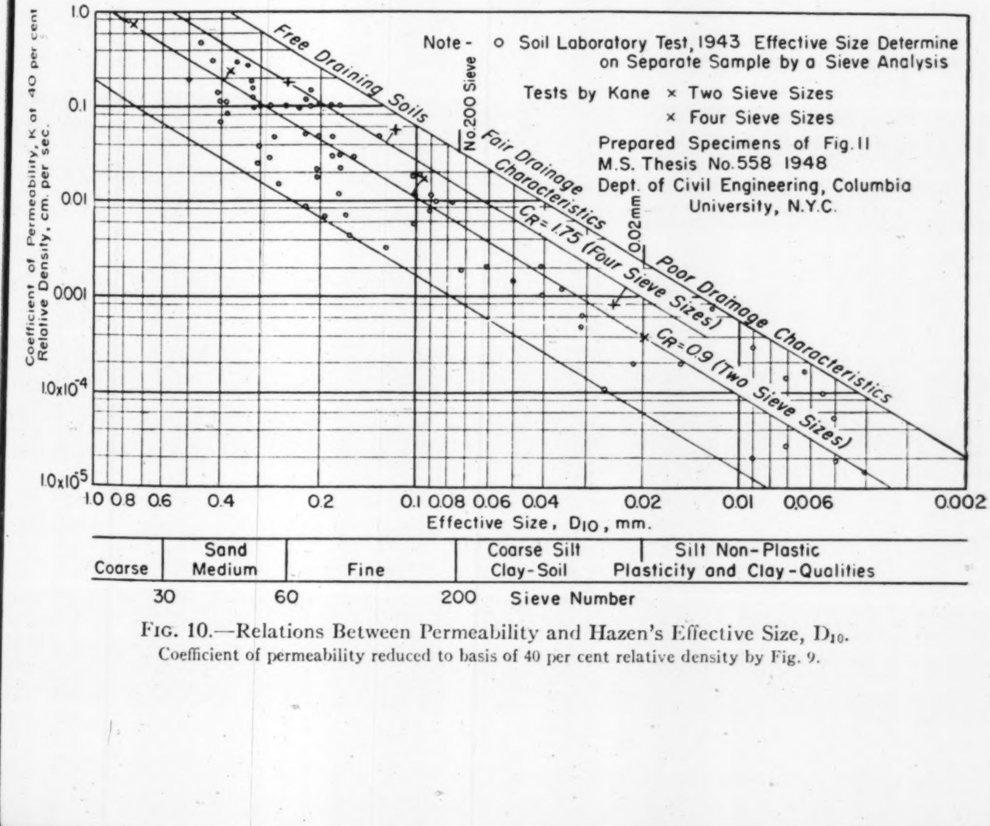


FIG. 10.—Relations Between Permeability and Hazen's Effective Size, D_{10}.
Coefficient of permeability reduced to basis of 40 per cent relative density by Fig. 9.

Permeability Relationships for Granular Soils:

In the design and construction of earth dams and of embankments used for impounding water, seepage may be of primary importance, not only through the embankment itself, but also through the abutment and foundation soils, particularly if they are of granular character. In order to obtain representative values of the coefficient of permeability that will bracket the limiting values corresponding to the probable range of field densities, it is necessary to obtain and to test a large number of undisturbed samples of granular soils. The problem would be greatly simplified if representative values of permeability could be obtained from tests on disturbed samples of granular soils and from field determinations of the probable range of field densities.

The possibilities of developing useful and practical permeability relationships with relative density are illustrated in Fig. 9. For each soil there are characteristic minimum and maximum densities and corresponding systems of void spaces and of soil channels through which water will flow, that controls permeability. Figure 9 shows that characteristic relationships exist between the logarithm of permeability and relative density, which is consistent with grain size distribution. The curves for the coarser grain soils with high permeabilities are relatively flat and almost linear with a small decrease in permeability with an increase in relative density. The curves for the finer-grained soils with lower permeabilities become steeper with a large decrease in permeability with increase in relative density, particularly from about 70 per cent relative density, where there is a marked characteristic curvature downward toward 100 per cent relative density. This is due to the clogging effect of the fine soil grains in the pore spaces with the close packing of the grain structure of the soil.

A number of important facts have been disclosed by the research of Kane.[9] First, the pattern of permeability relationships has been extended in Fig. 9, as noted, to show more definite relationships with grain size distribution. Each specimen of soil was initially completely saturated under full vacuum. Second, very good practical relationships of permeability with the effective size, D_{10}, were obtained,[10] as defined by the reference lines in Fig. 10. In order to have consistent relationships, the permeabilities were reduced to a common basis of 40 per cent relative density. This value was selected because so many natural deposits of granular soils are in the range of relative density slightly below 40 per cent. Third, Kane's research has shown that for all soils with an effective size larger than the No. 200 sieve Darcy's law is valid only for values of the hydraulic gradient less than about 0.3 for the loose state and about 0.6 for the dense state, which define about the upper limits of the region of laminar flow. Fourth, an important fact revealed by this research is that for partial saturation of granular soils under aerated conditions with air clogging, such as obtains in soils above the natural ground water level with the percolation of rain water downward, the permeability is from 40 to 50 per cent of that for the completely saturated condition. This tends toward the lower value for the finer soils. This is important information in estimating the infiltration capacity and the rate of drainage of surface soils in connection with the design of storm water drainage systems.

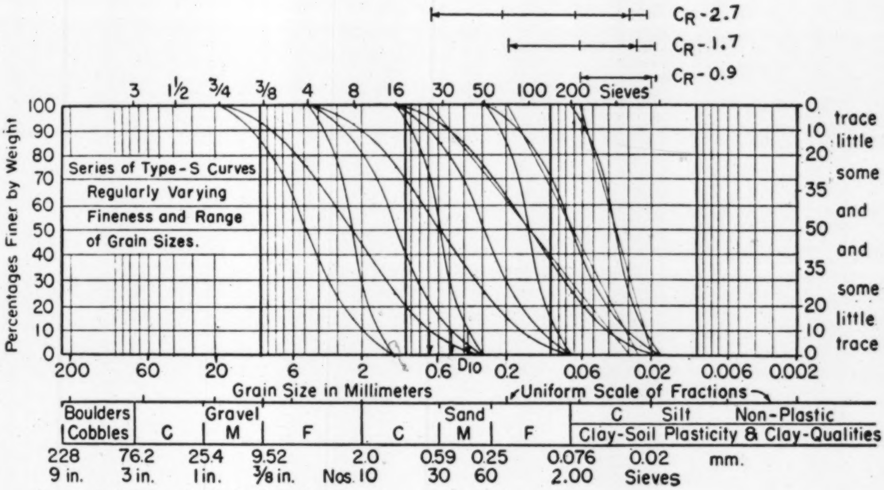
On the basis of the permeability curves

---

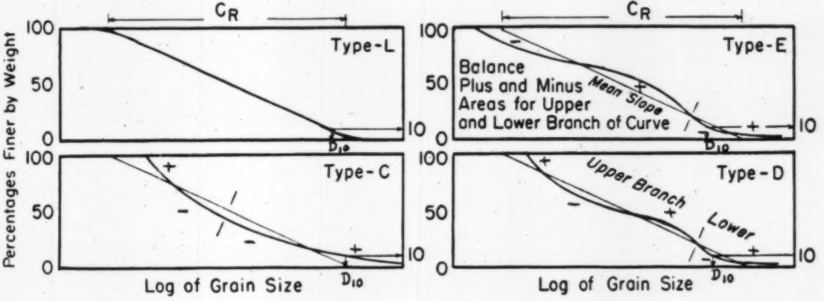
[9] H. Kane, "Investigation of the Permeability Characteristics of Sands," Thesis No. 558 for the M. S. Degree, Department of Civil Engineering, Columbia University, New York, N. Y., June, 1948. (Not published)

[10] Allen Hazen, *Annual Report*, Massachusetts State Board of Health, p. 539 (1892).

of Fig. 9, reasonably reliable estimates of permeability can be obtained covering the range of soils encountered on a project by making two permeability tests on each of a number of representative disturbed samples from each class of granular soil. The results of the two tests on each soil placed, for example, at 0 per sign purposes which will bracket the limiting values for a given situation. It is always desirable to make a few control tests on undisturbed samples of granular soils in the vertical and horizontal directions in order to determine the probable range of the ratio of the permeabilities in the vertical and horizontal directions.



(a) Type-S Grain Size Curves



(b) Type of Grain Size Curve

FIG. 11.—Distinguishing Characteristics of Grain Size Curves.
Fineness, range of grain sizes, and type.

cent and at 70 per cent relative densities will define a permeability curve, which can be drawn in general conformity with the pattern curves in the particular region of Fig. 9. By entering permeability curves with the range of field relative density determined for the given soil, reliable estimates can be obtained of the probable range of permeability for design purposes which will bracket the limiting values for a given situation.

A sufficient number of determinations of field relative densities should be made to bracket the limiting possibilities for a given situation.

In certain cases it is desirable to obtain preliminary information on the permeability of soils from grain size distribution curves. By taking the value of the permeability at 40 per cent relative density corresponding to the effective size, D_{10}, of a soil from the reference lines of Fig. 10, and by entering the permeability curves of Fig. 9, a permeability curve can be interpolated from this single point which conforms to the pattern of curves in this region. Estimates of the probable range of permeability can then be obtained for any range of relative density for the given soil.

GRADING-DENSITY RELATIONSHIPS

The foregoing relative density relationships show that grain size distribution, as the principal inherent characteristic of the granular soil material, plays an important rôle in shaping soil behavior. The maximum and minimum densities are characteristic and essential references for relative density for each individual soil, and determine the limits of their behavior, as illustrated in the foregoing diagrams. In order to express adequately the dependence of the maximum and minimum densities, and hence of behavior upon grain size distribution, the identifying and distinguishing characteristics of grain size distributions must be defined on an adequate and significant basis.

The three distinguishing characteristics of grain size distributions of granular soils are: fineness, range of grain sizes, and type of grain size curve,[11] which are entirely independent characteristics, as illustrated in Fig. 11. The fineness of a soil places a grain size distribution curve at a particular point on the scale of grain sizes. Hazen's[10] effective size, D_{10} the grain size for which 10 per cent of material is finer), has been widely used for identifying the fineness of soils, and is a satisfactory fineness index, as shown by the permeability relationships in Fig. 10. The range of grain sizes, in order to be of practical value for establishing significant relationships, must be representative of the entire grain size distribution curve for all types of curves. The range of grain sizes, designated C_R, is defined on a simple but satisfactory basis of the "mean slope" of the grain size curve. The mean slope is readily determined graphically, using a transparent scale, by making the plus and minus areas enclosed between the grain size curve and the mean slope equal and balanced independently for the upper and the lower branches of the grain size curve. This is shown in Fig. 11 for three types of curves. The range of grain sizes is then defined in units of the coarse, medium, and fine fractions of soil for convenience as noted in Fig. 11, or as twice the logarithm of the ratio of the grain sizes at the 100 per cent and 0 per cent terminal points of the mean slope:

$$\text{Range of grain sizes, } C_R = 2 \log \left(\frac{D_{100}}{D_0} \right) \quad . . (7)$$

This definition makes value of C_R equal to 1.0 for the coarse, medium, and fine fractions of soil (the simple ratio of the upper and lower grain size limits of these fractions being equal to 3.0). This definition of the range of grain sizes is significant because for the type-S grain size curve a linear relationship exists between C_R and the "standard deviation" commonly used in statistical analyses.

TABLE II.—TYPES OF GRAIN SIZE CURVES.

Type Designation	Description
S..........	S-shaped and practically symmetrical.
L..........	Linear.
C..........	Concave up and skewed to the coarser sizes. Predominantly coarse and tapering off gradually toward the finer sizes.
E..........	Convex up and skewed to the finer sizes. Predominantly fine and tapering off gradually toward the coarser sizes.
D..........	Irregular distributions with a hump, indicating a mixture of coarse and fine soils, which are typical of glacial deposits.

---

[11] D. M. Burmister, "Practical Methods for the Classification of Soils," *Proceedings*, Purdue Conference on Soil Mechanics and Foundation Engineering, September 2-6, p. 129, 1940.

The type or shape of grain size curve is an index of the symmetry or of the type of "skew" in the distribution of grain sizes. For example, the type-S grain size curve has the same symmetrical form as the curve of the normal law of chance used in statistical analyses. The types of curves are given letter designations, which are sufficient for practical purposes in soil analyses, as given in Table II and shown in Fig. 11. These types of curves are significant, because the geological processes operating to form soil deposits tend to produce characteristic types of grain size distributions. For example, sands deposited by the processes of sedimentation in flowing water and in quiet water, by wave action, and by wind action, have characteristic type-S grain size curves. For this reason the type-S grain size distributions in Fig. 11 were made up for the foregoing relative density studies, covering a range of grain size distributions commonly found in natural deposits from coarse silt to fine gravel by suitable steps of sieve sizes.

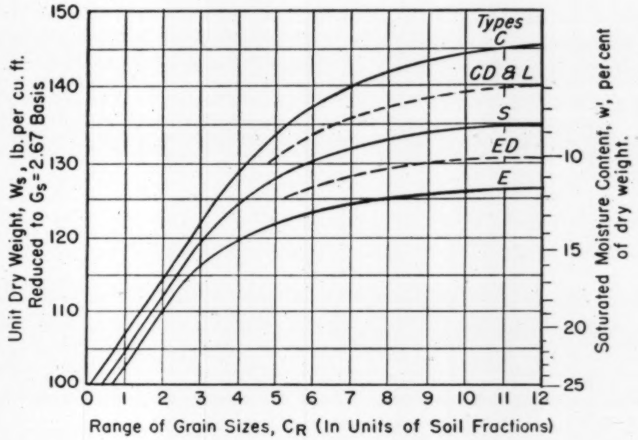


FIG. 12.—Maximum Compacted Densities. 100 per cent Relative Density.

Basic Physical Law Governing the Grading-Density Relationships for Granular Soils and for Clay-Soils of Less than Low (+) Plasticity. Maximum Densities Obtained by Vibrational Methods.

(a) Approximate minimum densities, 0 per cent relative density.

Range of grain sizes, C_R	Decrease in density, lb. per cu. ft. Coarser Soils		Finer Soils
1 to 3	15	to	20
3 to 5	20	to	25
5 and greater	25	to	30+

(b) Approximate influence of grain shape on density.

Grain shape	Change in density, lb. per cu. ft.
Very angular	−10 to −15
Sub-angular	0 Normal
Rounded or waterworn	+2 to +5
0.5 per cent mica	−2 to −5

On the basis of these characteristics of the grain size distributions of soils, grading-density relationships[12] have been established which show that the maximum and minimum densities are not haphazard phenomena but are controlled by definite physical laws. The grading-density relationships are presented in Fig. 12 for the maximum density as the 100 per cent reference for relative density. It is important to note that the range of grain sizes is the dominating physical chaacteristic of grain size distributions to a value of about 3.0, which includes the predominantly sandy soils. For values of the range of grain size greater than 3.0 the type of grain size curve becomes equally important in determining the maximum density. The "well-graded" soils with the ideal C-type gradings have the highest maximum densities. The maximum density of granular soils is practically independent of the fineness or coarseness of the soil.

---

[12] D. M. Burmister, "The Grading-Density Relations of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 587 (1938).

The minimum density, as the 0 per cent reference for relative density, is considerably affected by the fineness of the soil. Estimates of the approximate minimum densities can be obtained by deducting the value given by (*a*) and (*b*) of Fig. 12, from the maximum densities.

Since the maximum and minimum densities, as references for relative density and as characteristic states of the grain structure, determine the limits of the behavior of granular soils and are dependent upon grain size distribution, it is to be expected that grain size distribution will play an important and a determinable rôle in shaping the behavior characteristics of granular soils. The grading-density relationships and the relative density relationships greatly increase the possibilities for the practical use of grain size distribution curves of granular soils. A few detailed control tests to determine the strength properties of selected samples of soil near the upper and lower grain size distribution limits of each class of soils will serve as basis for making reliable estimates to cover the entire range of soils for each class of soils. Estimates of the maximum and minimum densities can be obtained from Fig. 12, which are sufficiently reliable for many practical purposes, usually within plus or minus 1 or 2 lb. per cu. ft. Estimates of the behavior characteristics can then be obtained from relative density relationships such as given herein, when completely developed, to cover the range of field relative densities for each class of soils. Suitable values of the behavior characteristics can then be selected which bracket the limiting values controlling a given situation.

CONCLUSIONS

Relative density as a fundamental concept and as a significant state of the grain structure of granular soils thus plays an important rôle in soil mechanics in bringing complex phenomena of soil behavior together on a common basis and in establishing basic physical relationships that control soil behavior. The cases cited illustrate, but by no means exhaust, the possibilities of developing completely basic physical relationships governing the behavior characterictics of soils, which can be made to cover practical ranges of grain size distributions of granular soils.

MEASUREMENT OF THE REACTIVITY OF SOLID FUELS BY THE CROSSING-POINT METHOD*

By J. Jonakin,[1] P. Cohen,[1] R. Corey,[1] and B. Jain[1]

Synopsis

A critical study has been made of the crossing-point method for determining the reactivity of solid fuels, and a modification developed in this investigation has been found suitable for measuring the adiabatic heating rates of small sizes of fuels. Tests of a Pittsburgh coal, an Indiana No. 5 coal, and a 900 C. experimental coke showed that (1) the order of the reaction with respect to oxygen was less than one, that of the lower rank, Indiana No. 5, being lowest; and (2) the heating rate per unit surface varied only slightly with the sieve size of the Pittsburgh coal and coke, but considerably with that of the coarsely-banded Indiana coal.

Increased understanding of the complicated technical processes of combustion and gasification must ultimately be acquired by correlating observations of these processes with *fundamental* properties of fuels. Of these properties, probably none is more important, nor has its quantitative evaluation been the subject of more study, than the reactivity, which may be defined as the rate of reaction of fuel and oxidizing gas, when determined under conditions such that it is a function of the properties of the fuel and not of the mass transport of reactant and product gases.

Since for each quantity of material reacted a definite quantity of heat is released, the rate of heat production may be taken as a measure of the reactivity for any given fuel. Although the proportionality between amount of heat released and quantity of material reacted is not a constant for all solid fuels, the variation is not great, and the rate of heat production may be taken as a comparative measure of reactivity for all solid fuels.

Under adiabatic conditions the heat produced by the reaction is used entirely to raise the temperature of the reacting system. With materials of high thermal diffusivity, or generally at very low rates of heating, each particle of the fuel at any instant will be at a substantially uniform temperature throughout. The observed rate of heating will then be equal to the rate of heat release divided by the heat capacity of the system. For the types of materials and heating rates that are of interest, this relationship might be questioned. However it can readily be demonstrated that at the critical point in the test procedure, the average temperature of particles of solid fuels of the sizes used in these tests is insignificantly lower than the surface temperature, and the relationship between heat release and rate of rise of surface temperature given above applies. In general, then, the rel-

---

* Presented at the Fifty-first Annual Meeting of the Society, June 21–25, 1948.

[1] Fuel Chemist, Fuel Engineer, Supervising Engineer, and Government of India Scholar, respectively, Combustion Research Section, Bureau of Mines, Central Experiment Station, Pittsburgh, Pa.

ative rates of reaction of identical size fractions of solid fuels having similar physical properties may be taken as the ratios of the observed adiabatic rates of change of temperature of the respective reacting systems. For some applications, such as the heating of coal in storage, the adiabatic rates of heating may be of direct interest.

Similar considerations led Sebastian and Mayers **(1)**[2] of the Coal Research Laboratory of the Carnegie Institute of Technology, to modify procedures previously developed for "ignition point" measurements so as to determine directly the relative reactivity of cokes. The method used is briefly described as follows: Air or oxygen is passed at a fixed rate through a bed of the fuel supported on a metal gauze in a glass tube, which is contained in a metal-block furnace heated at a constant rate. Measurements are made at uniform intervals of the temperature of the sample by means of a thermocouple with the junction at the center, and of the temperature of the furnace by means of a thermocouple with the junction in contact with the outer wall of the glass tube, and are continued until after the temperature of the sample exceeds the temperature of the furnace. At the instant that the sample and the furnace are at the same temperature, called the *crossing point*, it is assumed that there is no heat flow to or from the sample; the sample is heating adiabatically, and the rate of heating is a measure of the reactivity of the fuel.

Sebastian and Mayers listed a number of requirements for an apparatus in which adiabatic rates of heating could be determined and showed that their results for cokes could be correlated by an Arrhenius type equation:

$$\log \frac{1}{p}\frac{dT}{dt} = b - \frac{E}{4.575T} \quad \ldots\ldots\ldots \ (\mathbf{1})$$

where:

p = partial pressure of oxygen in atmospheres,

$\frac{dT}{dt}$ = heating rate of the sample at the crossing-point in degrees Centigrade per minute.

b = empirical constant,

E = activation energy in calories per gram mole,

T = absolute temperature of the sample at the crossing-point in degrees Kelvin, and

t = time in minutes.

From the crossing points and corresponding heating rates in oxygen and in air, the values are obtained from Eq. 1 for the temperatures at which the heating rates in oxygen would be 15 and 75 C. per minute. These temperatures, designated t_{15} and t_{75}, respectively, are defined as the *reactivity indices* of the fuel tested.

Sherman, Pilcher, and Ostborg **(2)** described an extended investigation of the Coal Research Laboratory Method, as developed by Sebastian and Mayers **(1)** and concluded that "it is well conceived to determine the temperature at which the rate of oxidation attains some definite value under standard conditions." In a subsequent publication from the Coal Research Laboratory, Orning **(3)** presented the results of tests by this method for a large number of coals and additional data for cokes. He correlated the reactivity indices with the proximate analysis of the fuels, noting, however, that the details of the apparatus and the thermometric procedure affect the absolute scale of the results. Also, on the basis of preliminary data from a new type of furnace, he stated that the order of the reaction with oxygen is more nearly zero for lignite, and increases toward one, with decrease in volatile content. This finding was confirmed in the subsequent investigation with the new apparatus recently reported by Orning, Mallov, and Neff (4).

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1289.

Work on the Coal Research Laboratory reactivity test was begun before the war by the Combustion Research Section of the U. S. Bureau of Mines to provide a basis for the correlation of fuel reactivity with combustion studies, but had to be suspended until recently. Upon resumption of this investigation, evidence accumulated which tended to show that some of the basic assumptions made by Sebastian and Mayers (1) in the design of the method for testing cokes, and verified by Sherman, Pilcher, and Ostborg (2) for bituminous coals, might not be correct. Before proceeding with systematic testing, the fundamentals of the method were re-examined.

Using an apparatus slightly modified from that of Sherman, Pilcher, and Ostborg (2), studies were made of: (*a*) the temperature distribution in the sample, near the crossing-point, to permit a rational location of the thermocouples; (*b*) the effect of gas composition; (*c*) the effect of rate of flow of gases; and (*d*) the effect of the size consist and method of preparation of the test sample. In addition, because of its critical effect on the determination, considerable attention was devoted to the calibration of thermocouples and the technique of potentiometric measurements. Although the work reported here is restricted to two bituminous coals and one coke, the results obtained appear to have quite general application. The apparatus and procedure developed are believed to give absolute values for the adiabatic rates of heating of the fuels tested, in the range for which the apparatus is suited.

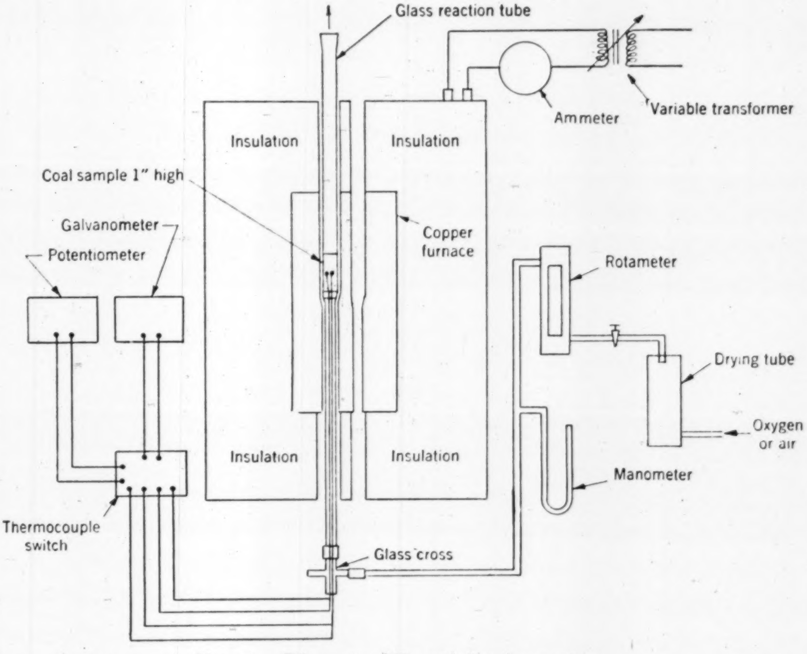

Glass reaction tube
Ammeter
Variable transformer
Insulation
Insulation
Coal sample 1" high
Galvanometer
Potentiometer
Copper furnace
Rotameter
Drying tube
Oxygen or air
Insulation
Insulation
Manometer
Thermocouple switch
Glass cross

FIG. 1.—Diagram of Reactivity Apparatus.

Apparatus:

Except for modifications in details, the apparatus used is essentially that described by Sherman, Pilcher, and Ostborg (2). The final assembly is shown in Fig. 1. The significant changes are as follows:

1. The copper cylinder, shown in Fig. 2, is drilled to permit circulation of water between tests, for rapid cooling of the furnace, thus saving considerable time.

2. The furnace is wound with 36 turns of No. 16 nichrome-V wire, having a total resistance of 10 ohms, and operates at 115 v. From each end to the center, each successive inch carries 7, 6, and 5 turns of wire, respectively, to compensate for the end losses.

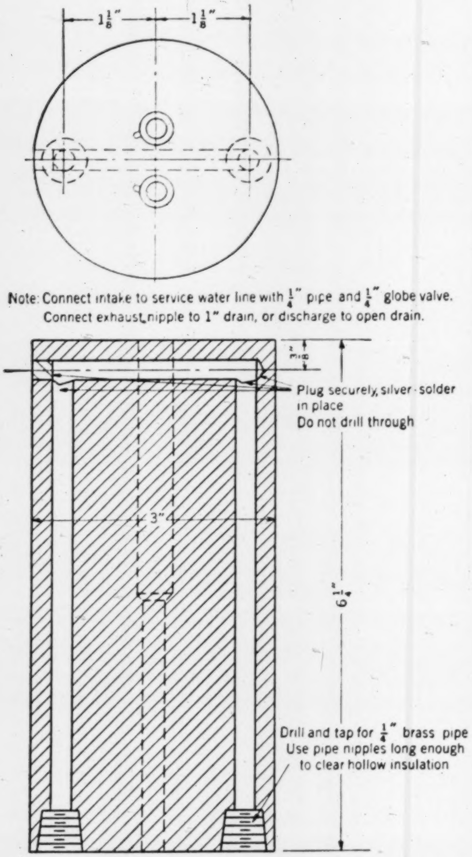

$1\frac{1}{8}''$
$1\frac{1}{8}''$
Note: Connect intake to service water line with $\frac{1}{4}''$ pipe and $\frac{1}{4}''$ globe valve.
Connect exhaust nipple to 1" drain, or discharge to open drain.
Plug securely, silver-solder in place
Do not drill through
3"
$6\frac{1}{4}''$
Drill and tap for $\frac{1}{4}''$ brass pipe
Use pipe nipples long enough to clear hollow insulation

FIG. 2.—Detail of Copper Block Showing Method of Water Cooling.

3. Since the temperature coefficient of resistance of the winding is quite low, the power input to the furnace is maintained sufficiently constant by operating at constant current, as indicated by an ammeter. This arrangement replaces the watt meter used by Sherman, Pilcher, and Ostborg (2).

4. As shown in Fig. 1, the apparatus is in its final form. There are two thermocouples in the sample—one at the axis, and one at a radial distance of 2 mm. (In the tests in which a furnace thermocouple was used, the arrangement was that of Sherman, Pilcher, and Ostborg (2).) The thermocouple insulating tube is 0.1 in. in outside diameter and had four 0.019-in. holes. The plug supporting the sample consists of a strip of 100 mesh nichrome gauze, $\frac{1}{4}$- in. wide and wrapped snugly around the thermocouple insulating tube, so that only $\frac{1}{16}$ in. of the tube projects beyond the upper surface of the plug. The thermocouples are No. 28 gage chromel-alumel, and $\frac{7}{16}$ in. of bare wire project beyond the end of the insulating tube, placing the junctions $\frac{1}{2}$ in. above the top of the gauze plug.

5. The thermocouple circuits and switching arrangements are shown in Fig. 3. The emf. of the center thermocouple is measured with a Leeds & Northrup Model 8662, Precision Portable potentiometer. The differential emf. between the center thermocouple and either a radial sample thermocouple or an external furnace thermocouple is measured with a Model 3414 Rubicon "spotlight" galvanometer, with a sensitivity of 10 microvolts per mm. scale deflection.

6. During the test, the sample is held in place and the loss of fines prevented by means of a nichrome gauze disk, 8 mm. in diameter, topped by a porcelain tube with an arbitrarily weighted extension.

7. A glass cross, with a rubber tubing connection to the bottom of the reactivity tube, is used to introduce the thermocouples at the bottom of the cross, the oxidizing gas at one side arm and compressed air, for blowing out the sam-

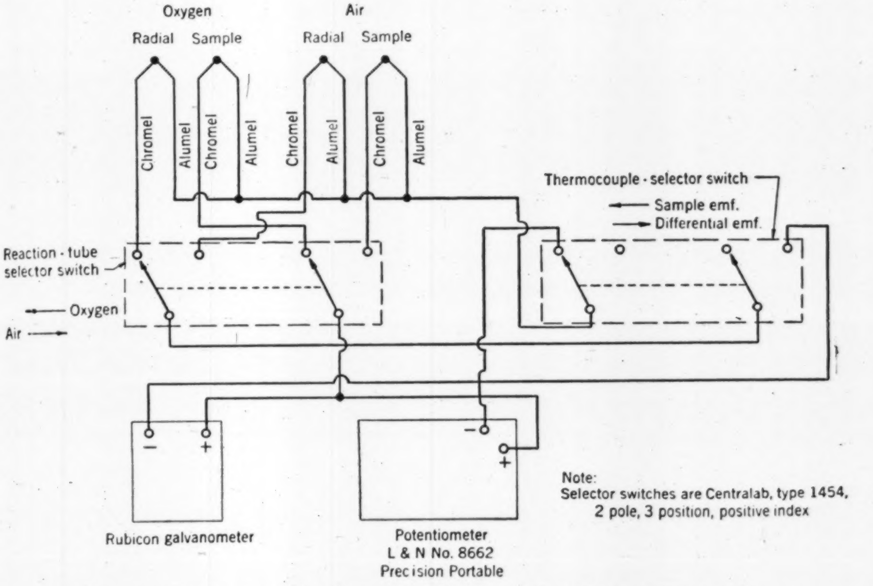

Oxygen
Radial
Sample
Air
Radial
Sample
Chromel
Alumel
Chromel
Alumel
Chromel
Alumel
Chromel
Alumel
Thermocouple - selector switch
Sample emf.
Differential emf.
Reaction - tube selector switch
Oxygen
Air
−
+
−
+
Rubicon galvanometer
Potentiometer
L & N No. 8662
Precision Portable
Note:
Selector switches are Centralab, type 1454,
2 pole, 3 position, positive index

FIG. 3.—Diagram of Thermocouple Wiring and Switching Arrangement.

TABLE I.—RECORD OF TYPICAL TEST DATA.

Test No....	25	Date.....	4–18–47
Sample.....	Warden Mine, Pgh. Bed	Size......	−40 + 60 mesh, stage-crushed
Current....	8.7 amps.	Weight...	0.900 g.
		Volts.....	75

Rate of Flow of Gas, cu. cm. per min.	Left tube	Right tube
Oxygen........................	65	..
Air............................	..	..

Time, min.	Emf. of Sample Couple, mv.	Emf. of Differential Couple, mv. (Radial T. C.—Sample T. C.)
16.00	7.38	
16.25		0.067
16.50	7.63	
16.75		0.055
17.00	7.88	
17.25		0.044
17.50	8.14	
17.75		0.029
18.00	8.41	
18.25		0.010
18.50	8.70	
18.75		−0.012
19.00	8.99	
19.25		−0.041
19.50	9.32	
19.75		−0.080
20.00	9.68	
20.25		−0.130
20.50	10.01	
20.75		

ple at the end of the test, at the other side arm. Air and oxygen, filtered and dried, are metered with calibrated rotameters. The static pressure in the tube is measured with a water manometer.

The reactivity tubes are of the same dimensions as those used by Sherman, Pilcher, and Ostborg (2). The bulbs at the top of the tube, and the constrictions and the ground glass joint at the bottom of the tube have been eliminated.

Test Procedure:

A weighed quantity of the sample sufficient to give a height of 1 in. is transferred to each reactivity tube, the tube is gently tapped, and the weight for holding the sample in place is applied. The reactivity tubes are lowered into the furnace, the flow of gases started and adjusted to the desired rate, and the furnace current is adjusted to give the desired heating rate. Readings of the emf. of the axial thermocouple and of the differential emf. between the two thermocouples are made alternately at 30-sec. intervals and are continued for 2 to 3 min. after the crossing point. The current to the furnace and the rates of flow of gases are maintained constant during the test. For the fuels so far tested, sufficient time has always been available to make the necessary measurements on the air sample after the oxygen test has been completed. At the close of the test the samples are blown out by compressed air, after first pinching off the gas inlet tube at the cross. The reactivity tubes are inspected, and if necessary, are carefully cleaned to remove all traces of sample.

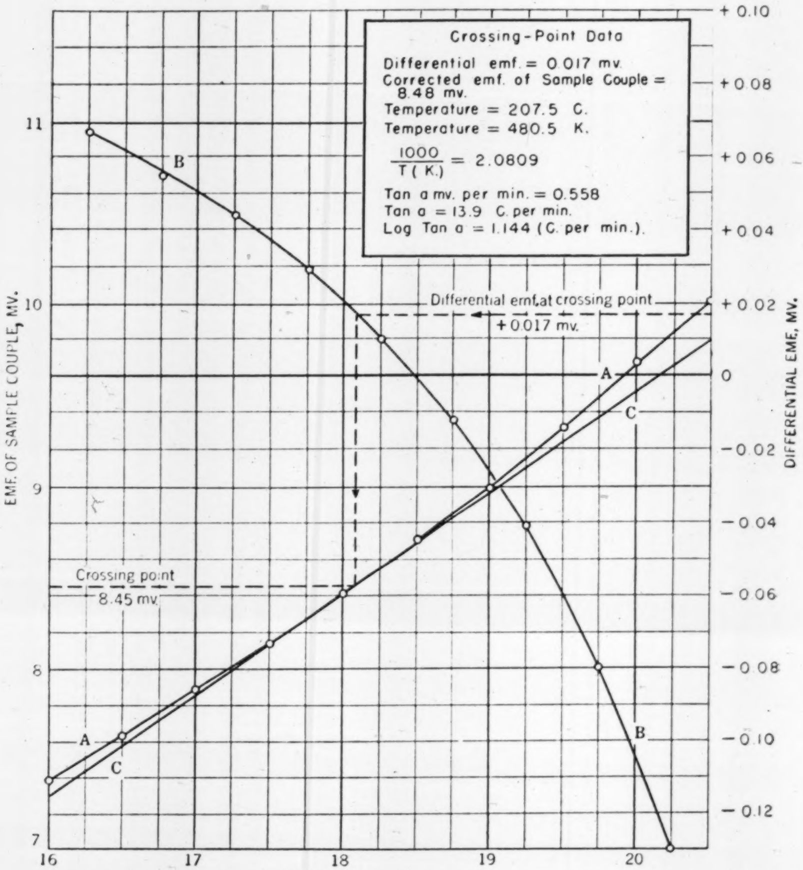


Fig. 4.—Graphical Determination of Crossing Point and Heating Rate.

The data recorded in a typical test with oxygen are given in Table I. Figure 4 shows how these data are plotted to obtain the crossing point and the corresponding heating rate. Curve *A* is the emf.-time curve for the axial or sample thermocouple, curve *B* is the differential emf.-time curve, and line *C* is the tangent to curve *A* at the crossing point. According to the differential calibration of the thermocouples, the temperature difference is zero when the differential emf. is 0.017 mv. The rate of change of the temperature of the sample at the crossing point is equal to the slope of line *C*, millivolt per minute, multiplied by the temperature coefficient of the thermo couple at the corresponding emf., degree Centigrade per millivolt.

Temperature Measurements—Thermocouple Calibration:

The determination of the crossing point, defined as the time when the axial (sample) thermocouple and a thermocouple at some other location are at the same temperature, is extremely sensitive to errors of measurement and calibration. If an error of 1 C. is made in the temperature difference of the thermocouples in the test illustrated in Table I and Fig. 4, which is equivalent to 0.04 mv., the resultant error in the determination of the crossing-point temperature will be 14 C. Errors of measurement are largely eliminated by the use of a differential method and a sensitive galvanometer. The reduction of errors of calibration is considerably more difficult to accomplish.

Chromel-alumel thermocouples are for several reasons the logical choice for this application. They have satisfactory sensitivity, are low in cost, and thus may be frequently replaced, and except for attack by sulfur, are quite resistant to corrosion. A considerable disadvantage, however, is the comparatively low homogeneity, with consequent errors in the presence of high temperature gradients. Thermocouples of this type must be calibrated in place, and the temperature gradients must either be low or a function only of the temperature to be measured.

After considerable preliminary study, the reactivity apparatus was critically tested to determine the limits of calibration possible for various locations of the thermocouples. For the usual thermocouple porcelains there were substituted special 6-hole insulating tubes of the same external diameter, in which were mounted, in addition to the chromel-alumel thermocouples, reference couples of copper-constantan, with an inhomogeneity error of less than one part in 5000. The copper-constantan couples, consisting of one No. 35 gage copper wire, and three No. 30 gage constantan wires, were tested for homogeneity by varying the depth of immersion of the couple in vapor baths of sulfur, naphthalene and benzophenone, boiling at atmospheric pressure, using the apparatus of Mueller and Burgess **(5)**. Comparisons were made between the corresponding chromel-alumel and copper-constantan thermocouples, in place in the furnace with normal air flow rates, and for furnace temperature constant, and increasing at 15 C. per min.

TABLE II.—COMPARISON OF CHROMEL-ALUMEL THERMOCOUPLES WITH COPPER-CONSTANTAN THERMOCOUPLES IN PLACE IN REACTIVITY FURNACE.

Thermocouple Position	Emf. of Reference Copper-Constantan Couple, mv.	Emf. of Cr-Al Couple, mv. Heating Rate of Furnace		Difference in emf., mv. (15 C. per min. — 0 C. per min.)
		15 C. per min.	0 C. per min.	
1	*2*	*3*	*4*	*5*
		Standard Oxidized Wire		
Furnace......	5.73	5.88	5.81	+0.07
Furnace......	9.33	8.86	8.81	+0.05
Sample......	5.71	5.78	5.79	−0.01
Sample......	9.33	8.82	8.81	+0.01
		Standard Oxidized Wire Annealed 40 hr. at 1000 F.		
Furnace......	13.22	11.99	12.01	−0.02
Sample......	13.26	12.08	12.07	+0.01

The results of this comparison are given in Table II. Column 1 of this table shows the location of the chromel-alumel thermocouple for which comparison is being made between the emf. at a heating rate of 15 C. per min., and at zero heating rate, at a temperature equivalent to a particular emf. of the corresponding copper-constantan thermocouple, as given in column 2. The values of the observed emf. of the chromel-alumel thermocouples are given in columns 3 and 4 for heating rates of 15 C. and 0 C. per min., respectively, and the difference in emf. in column 5. The data of the first four rows, for wire as-received from the manufacturer (standard oxidized wire), indicate that there is no significant difference in the emf. of the sample thermocouple, at a given temperature, when the heating rate is varied, whereas in the case of the furnace thermocouple, the variation in emf. is considerable. Thus, a calibration of the furnace thermocouple made at stationary furnace temperatures, would be in error by the amounts shown in column 5, if used at a rate of heating of the furnace of 15 C. per min. In constrast, the considerably smaller error for the sample thermocouple, ±0.01 mv., is noteworthy.

In an attempt to improve the homogeneity of the thermocouple, the standard oxidized wire was given an additional anneal of 40 hr. at 1000 F. Immersion tests in the sulfur vapor bath indicated that this treatment considerably improved the stability and homogeneity of the thermocouple. The last two rows of Table II show the results of tests on the specially treated thermocouples—a definite improvement is indicated. Subsequently, all thermocouple wire was given the additional treatment prior to use in the reactivity apparatus.

Having established that the calibration of the sample thermocouple (whether standard oxidized or specially annealed wire) is independent of the heating rate of the furnace, and that the same is true for specially annealed wire in the furnace location, the procedure was adopted of checking the thermocouples in place in the furnace against a standard platinum-rhodium, platinum thermocouple, calibrated at the boiling points of sulfur, naphthalene, and benzophenone. The standard thermocouple is supported from the top of the reactivity tube so that its junction is in contact with that of the sample thermocouple. A quantity of specially purified sand is then poured into the reactivity tube, sufficient to give a bed depth of 1 in. The furnace is allowed to come to constant temperature, and readings are then taken of the emfs. of the standard and sample thermocouples, and of the differential emf. of the reactivity thermocouples. This procedure is repeated at a number of temperatures, and smooth calibration curves are drawn.

Temperature Distribution in the Sample at the Crossing-Point; Location of the Thermocouples:

The thermocouples of the reactivity apparatus must be so located that at their crossing-point the portion of the sample adjacent to the sample thermocouple is heating adiabatically. As pointed out by Orning (3), this is probably not true for the location of the furnace thermocouple as used by Sebastian and Mayers (1) and Sherman, Pilcher, and Ostborg (2). Orning further concluded that the fraction of heat lost was relatively constant, thus resulting only in an error in the absolute scale of the reactivity indices. However, unless this fraction was the same for all reactivity tubes, even though it was independent of temperature and heating rate for any given tube, no use could be made of this characteristic.

To settle this point, tests were made on −40 + 60 mesh samples of Pittsburgh Bed, Warden Mine coal in air and in oxygen, with the furnace thermocouple outside the reactivity tube; both reactivity tubes were used for the tests with each oxidizing gas. The results of these tests, shown in Fig. 5 in the form of an Arrhenius plot, indicate that with this location of the thermocouple, the individual characteristics of the reactivity tubes are not eliminated, each tube yielding a

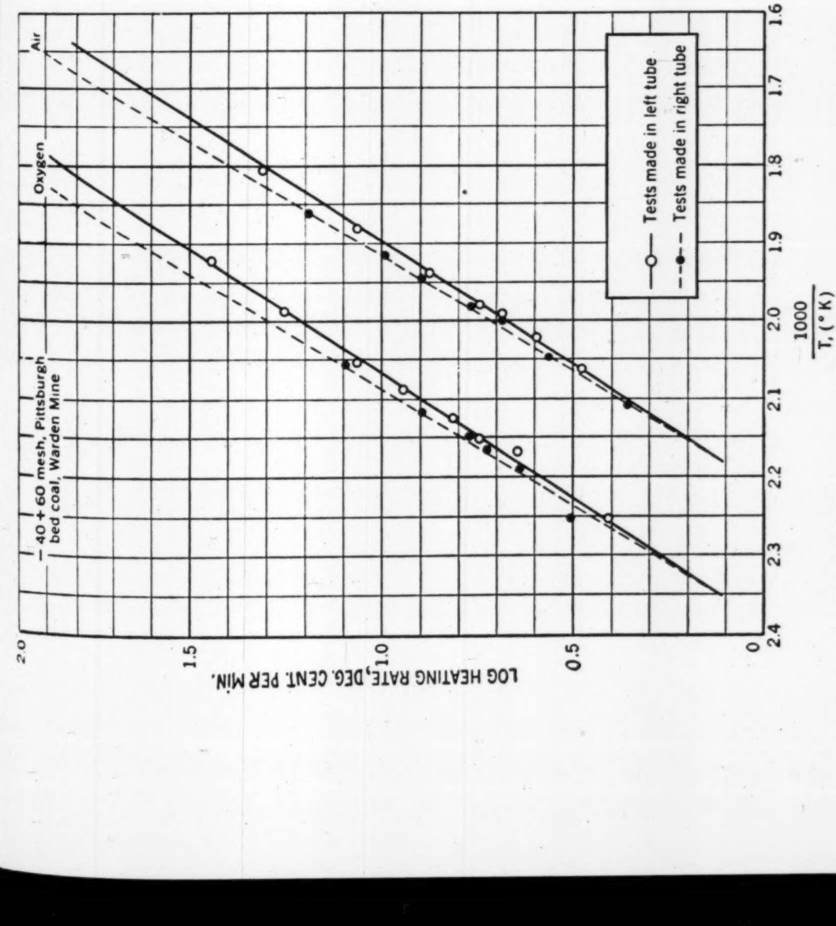


FIG. 5.—Effect of Different Tubes on Observed Heating Rate for Furnace Thermocouple Outside and Touching Tube.

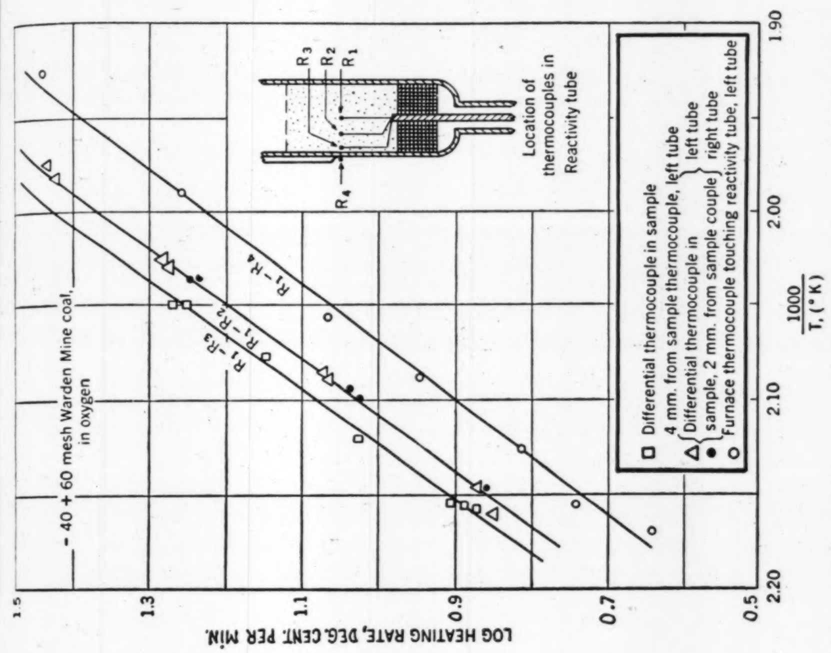


FIG. 6.—Effect of Thermocouple Location on Observed Heating Rate.

characteristic slope, which is the same for both oxygen and air. If, as had been done previously, all oxygen tests had been made in one tube, and all air tests in the other, entirely different and erroneous conclusions would have been drawn. Thus, if the oxygen tests had been made in the right tube only and the air tests in the left, the ratio of the rates of heating in oxygen and in air would have increased with temperature; whereas the other combination of tubes would have shown the opposite effect.

Tests were next made at various heating rates on Warden Mine coal in oxygen with a thermocouple at the center of the sample and differential thermocouples at 2 mm. and 4 mm. radial distances from the center or sample thermocouple. The results of these tests are shown in Fig. 6 in the form of an Arrhenius plot. For comparison, the corresponding data for the furnace thermocouple location have been included. It is seen that at any temperature, the heating rate is highest for the differential thermocouple in the sample, 4 mm. from the axis, R_3, slightly lower for the differential thermocouple in the sample 2 mm. from the axis, R_2, and considerably lower in the case of the furnace thermocouple, R_4. As noted on the figure, tests with the differential thermocouple located 2 mm. from the axis of the sample were made in both reactivity tubes. In contrast to the experience with the furnace thermocouple, the agreement of the two tubes is quite good, the average deviation of the results for the two tubes being only ±0.7 C.

Though the thermocouple located at R_3 yielded the highest heating rates, this in itself was no guarantee of adiabatic heating; it was quite possible that this location of the thermocouple corresponded to heat flow to the center of the sample at the crossing-point. The data accumulated in these tests were therefore analyzed to obtain the radial temperature distributions in the sample, at various significant times in a particular test, when the heating rate of the sample at the crossing-point of R_1 and R_2 was approximately 16 C. per minute. The results of this analysis are shown in Fig. 7, in which the temperatures at the midplane of the reactivity sample are plotted against the radial distance from the center of the sample, drawn to scale, for various times after the start of the test, including the crossing points of R_1 and R_3, R_1 and R_2, and R_1 and R_4. The

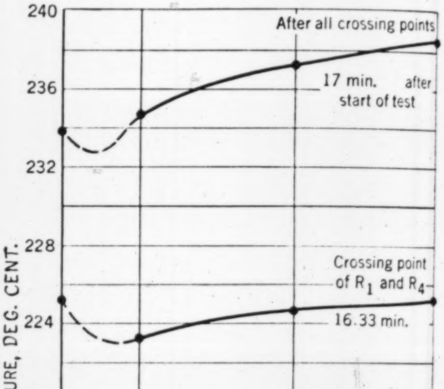


FIG. 7.—Temperature Distribution in the Sample During Reactivity Test.

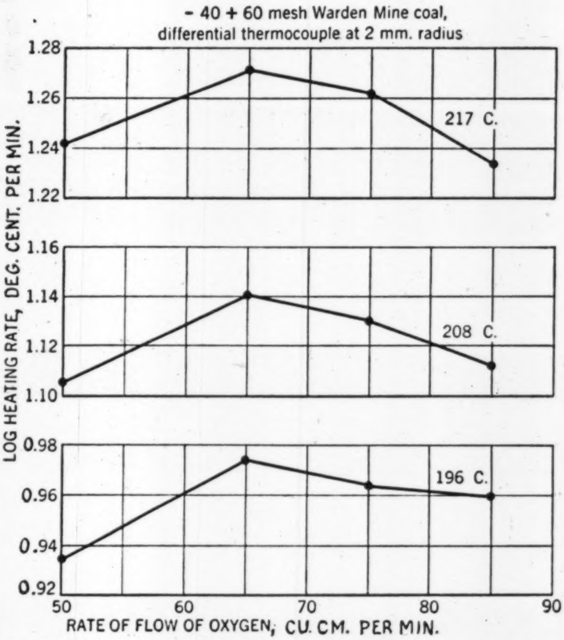


Fig. 8.—Effect of Rate of Flow of Oxygen on Heating Rate at Constant Temperature.

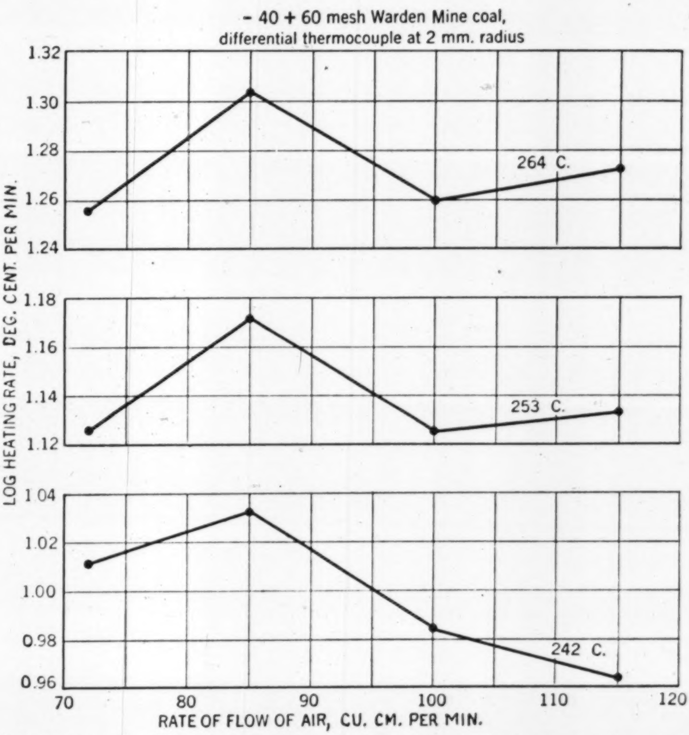


Fig. 9.—Effect of Rate of Flow of Air on Heating Rate at Constant Temperature.

dashed extensions of the curves show the *probable* temperature distributions in the wall of the reactivity tube. These curves are in complete accord with the results of Fig 6. Thus, at the crossing point of R_1 and R_3 some heat is flowing to the center of the sample; at the crossing point of R_1 and R_2 heat flow is negligible, and at the crossing point of R_1 and R_4 there is a considerable heat flow from the center of the sample to the wall.

It is concluded from these tests that a differential thermocouple in the sample, 2 mm. radially distant from the axis, yields results most closely approximating adiabatic heating. It is also evident from Figs. 6 and 7 that the position of the radial thermocouple is not critical; that is, small variations will have negligible effect. In all subsequent tests, therefore, the crossing-point was determined with a differential thermocouple of bare wire at the 2-mm. position, R_2, and a bare wire axial thermocouple, R_1, installed as shown in Fig. 6.

Effect of Rate of Flow of Gases on Heating Rate:

Since minimum crossing-point temperatures were obtained by Sebastian and Mayers (1) with cokes, and Sherman, Pilcher, and Ostborg (2) with Pittsburgh Bed coal, at rates of flow of 50 and 85 cu. cm. per min. in oxygen and air, respectively, these rates of flow were used in this investigation in the tests so far described. However, the crossing-point temperature alone is not an adequate indication of the behavior of the system. What is required, in this instance, is the effect of rate of flow of gases on the heating rate of the sample at various fixed temperatures. Tests were made, therefore, with varying rates of flow of air and oxygen for samples of a −40 + 60 mesh fraction of Pittsburgh Bed Coal, with a differential thermocouple at a radial distance of 2 mm. from the axis of the sample. The results with oxygen are shown in Fig. 8, and with air in Fig. 9. In each plot there is shown the variation of log sample heating rate with rate of flow of the gases, for three temperatures. The curves have well defined maxima at 65 cu. cm. per min. and 85 cu. cm. per min. for oxygen and for air, respectively, but the shapes of the curves do not conform well with the results previously obtained by Sebastian and Mayers (1) and Sherman, Pilcher, and Ostborg (2). Fifty-three tests were made in this phase of the investigation, and no evidence was found to indicate that the results obtained were conditioned by experimental factors not previously considered. Additional tests with an Indiana coal in oxygen showed no variation of log heating rate with rate of flow of gas at constant temperature for the entire temperature range studied. As a result of this study, standard rates of flow of 65 cu. cm. per min. for oxygen and 85 cu. cm. per min. for air were used in all subsequent tests.

Effect of Partial Pressure of Oxygen on Heating Rate. Order of Reaction:

The data of this investigation fail to confirm the findings of Sebastian and Mayers (1) for coke, and Sherman, Pilcher, and Ostborg (2) for bituminous coals, that the rate of heating at constant temperature is proportional to the partial pressure of the oxygen in the gas, as shown by Eq. 1. On the Arrhenius plots used here, this would require that the logarithms of the heating rates in oxygen and in air, at constant temperature, differ by 0.678, which is the difference of the logarithms of the partial pressure of oxygen in pure oxygen and in air. That this is not true for the fuels tested in this investigation is evident from Figs. 10, 11, 12, and 13. Figure 10 is an Arrhenius plot of the results of tests in oxygen and in air, of −20 +40 −40 +60, and

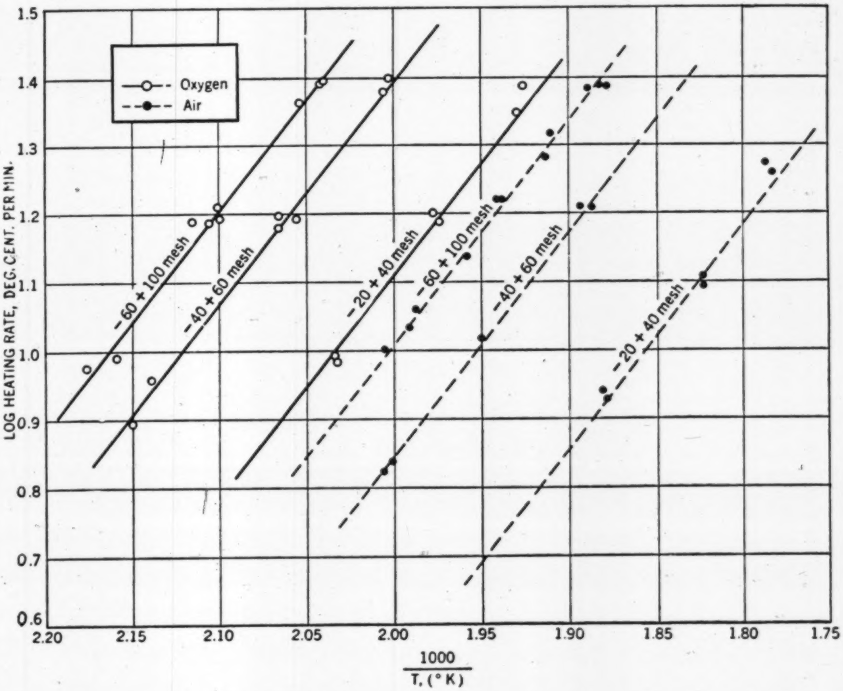


FIG. 10.—Variation of Heating Rate with Temperature, in Air and in Oxygen, of Stage-Crushed Samples, Pittsburgh Bed, Warden Mine Coal.

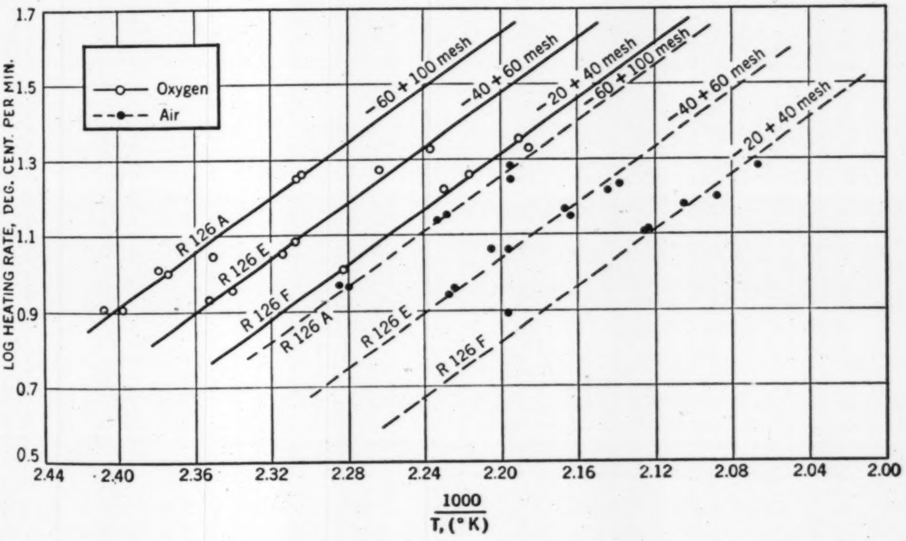


FIG. 11.—Variation of Heating Rate with Temperature, in Air and in Oxygen, of Stage-Crushed Samples, Indiana No. 5 Coal, New Hope Mine.

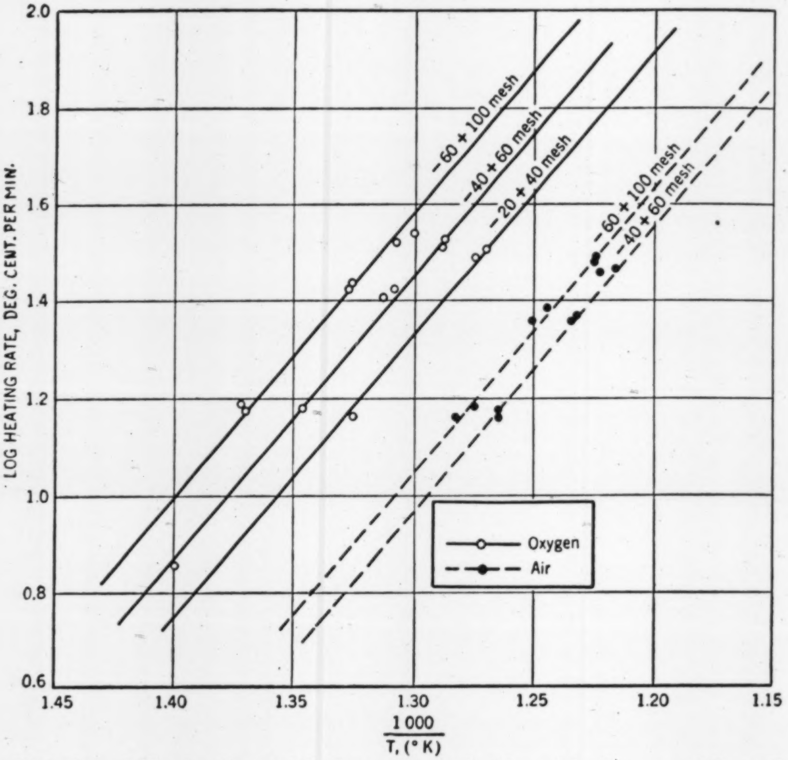


FIG. 12.—Variation of Heating Rate with Temperature, in Air and in Oxygen, of Stage-Crushed Samples of 900 C. Experimental Coke from Upper Elkhorn Coal.

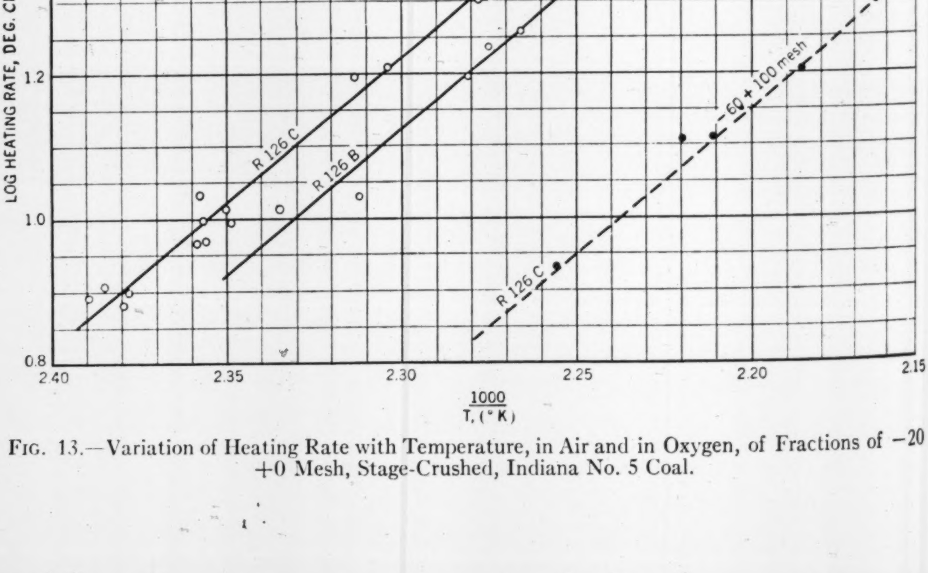


FIG. 13.—Variation of Heating Rate with Temperature, in Air and in Oxygen, of Fractions of −20 +0 Mesh, Stage-Crushed, Indiana No. 5 Coal.

−60 +100 mesh, stage crushed samples of Pittsburgh Bed, Warden Mine coal, and Figs. 11 and 12 are similar plots for Indiana No. 5 Bed, New Hope Mine coal, and a 900 C. experimental coke from Upper Elkhorn coal, respectively. Figure 13 is a similar plot of results of tests with other fractions of Indiana No. 5 Bed, New Hope Mine coal. For these three fuels, the variation of rate of heating with temperature is well represented by the Arrhenius law, but the pressure dependence of the reaction, though reasonably independent of temperature and the size-consist of the samples in the range of variables studied, is less than that postulated in Eq. 1.

TABLE III.—APPARENT ORDER OF REACTION WITH OXYGEN FOR STAGE CRUSHED SAMPLES OF PITTSBURGH BED COAL, INDIANA NO. 5 BED COAL, AND 900 C. EXPERIMENTAL COKE.

Fuel	Sample		Proximate Analysis, Parent Sample, per cent			Temperature,[a] deg. Cent.	Apparent Order of Reaction	
	Size, mesh	Designation	Fixed Carbon	Volatile Matter	Ash		Observed	Calculated Eq. 3
Pittsburgh Bed, Warden Mine Coal			57.2	36.4	6.4			
	−20 +40					251	0.86	0.79
	−40 +60					227	0.81	
	−60 +100					217	0.77	
Indiana No. 5 Bed, New Hope Mine Coal			45.6	42.9	11.5			
	−20 +40	R126A				187	0.72	0.72
	−40 +60	R126E				177	0.65	
	−60 +100	R126F				169	0.56	
900 C. Experimental Coke from Upper Elkhorn Coal			91.9	1.8	6.3			
	−20 +40					b	b	0.95
	−40 +60					492	0.71	
	−60 +100					479	0.78	

[a] Temperature at which rate in O_2 = 25 C. per min. Corresponds approximately to midpoint between highest temperature of test in O_2 and lowest temperature of test in air.

[b] No data were obtained in air for this size.

It will be noted in these figures that in most cases there is a slight gap between the highest temperature at which tests were made in oxygen and the lowest temperatures at which tests were made in air for the same sample. Thus, strictly speaking, the effect of oxygen pressure on heating rate has not been determined experimentally at any given temperature. However, generally excellent conformity of the test data with the Arrhenius law, and the short extrapolations required to reach a common temperature, leave little doubt of the validity of the calculated ratios of the rates of reaction in oxygen and in air for this temperature region.

The apparent orders of reaction with oxygen, expressed as

$$n = \frac{\log\left(\dfrac{\text{Heating rate in oxygen}}{\text{Heating rate in air}}\right)}{\log\left(\dfrac{\text{Pressure of } O_2 \text{ in oxygen}}{\text{Pressure of } O_2 \text{ in air}}\right)} \quad \ldots . \text{ (2)}$$

for the various stage-crushed samples of the three fuels tested, are summarized in Table III, which also includes the proximate analyses of the parent samples. For the two coals, the order of the reaction decreases with increasing fineness of the sample, and the relative values are in line with the rank of the coal, the Indiana No. 5 Bed coal having a lower order of reaction than the Pittsburgh Bed coal.

In their recent paper, Orning, Mallov, and Neff (4) have reported the results of the determination of the heating rate of solid fuels in an apparatus designed to permit the sample to heat adiabatically continuously. They found that although the observed ratios of the heating rates in oxygen and in air showed no significant trends with rank of coal at constant temperature, the ratios tended to increase with increasing temperature. An approximate relationship was derived, given by the equation:

$$R = 4.76 - 16e^{-\frac{T}{200}} \quad \ldots\ldots\ldots (3)$$

where:

T = absolute temperature in degrees Kalvin, and

R = the ratio of the rates of heating in oxygen and air.

Since the range of heating rates over which measurements can be made is quite small and independent of the reactivity, the temperature range over which measurements are made is also quite small, and the temperature level of measurement varies with the reactivity of the sample. Thus, Eq. 3 can conceivably be a relationship between order of reaction, and reactivity as expressed by the temperature at which the heating rate is in the measurable range in both gases; in any comparison such a temperature must be used. Comparison of the results of this investigation with those of Orning, Mallov, and Neff **(4)**, as shown by the calculated values in the last column of Table III, indicates fair agreement except for the coke.

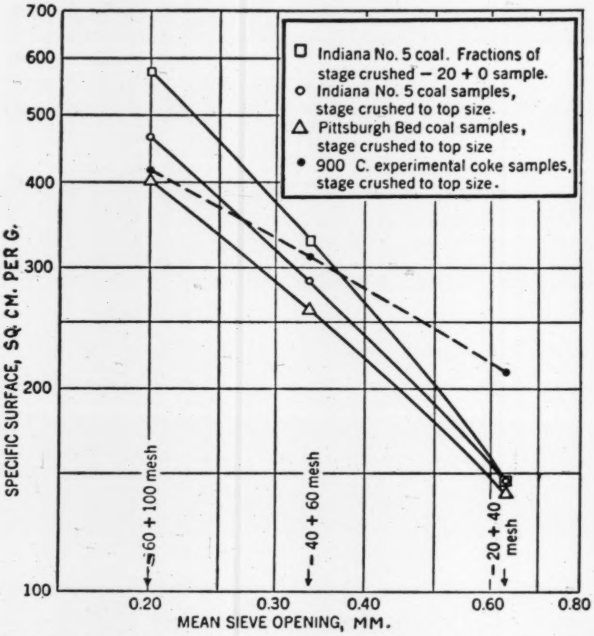


FIG. 14.—Specific Surface of Various Sizes of Coals and Coke Used in Tests.

Effect of Size Consist of Fuel on Reactivity; Preparation of Samples:

The rate of reaction of fuels with oxygen is markedly affected by the size of the fuel. In the absence of mass transfer controls, the rate of reaction must be proportional to the surface of the sample, if the reactivity per unit surface is independent of size. In evaluating the relative reactivity of different fuels, care must be exercised in the interpretation of the results to separate the effects of

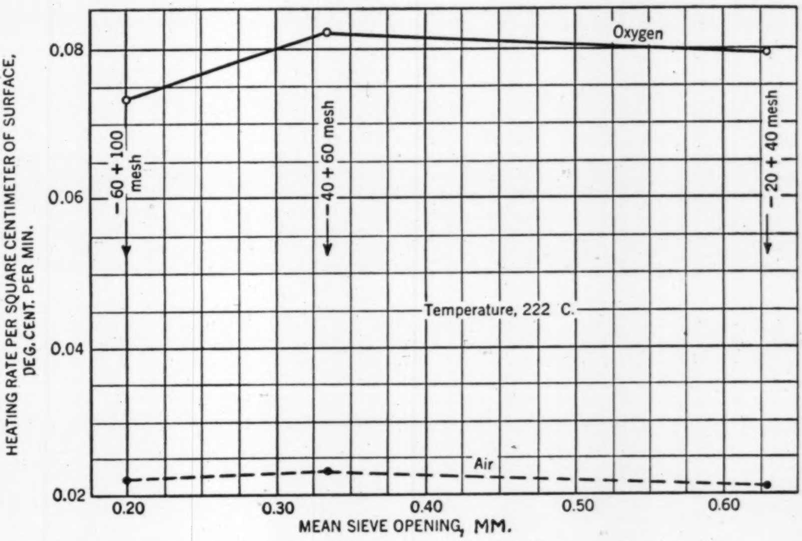


FIG. 15.—Effect of Size on Reactivity per Unit Surface of Pittsburgh Bed Coal, Warden Mine.

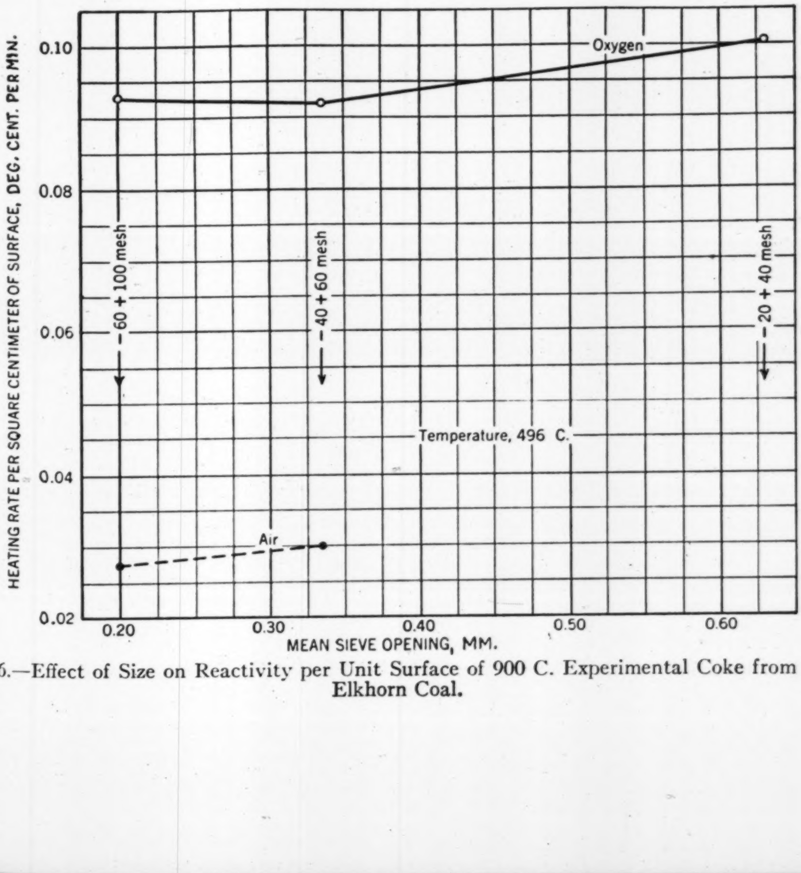


FIG. 16.—Effect of Size on Reactivity per Unit Surface of 900 C. Experimental Coke from Upper Elkhorn Coal.

reactivity on the one hand and specific surface on the other. For the sake of convenience in operation, and for uniformity in preparation, stage-crushed −40 +60 mesh samples have been used for most of the comparative studies of the reactivity of fuels. That the specific surface of this size of fuel can vary considerably is shown by Fig. 14, in which the specific surfaces of the various size fractions of the fuels tested have been plotted against the mean sieve opening of the sample. The specific surfaces were determined by a modified Lea-Nurse (6) air-permeability method. Thus, the specific surface varies from 285 sq. cm. per g. for −40 +60 mesh stage-crushed Warden Mine coal, to 328 sq. cm. per g. for the −40 +60 mesh fraction of −20 mesh New Hope Mine coal.

For a constant shape factor, the specific external surface will vary as the reciprocal of the particle size, and on the log plot of Fig. 14, a slope of −1 should result. Of the three series of coal samples, the fractions of the −20 +0 mesh Indiana No. 5 coal conformed most closely to this condition. The curve for the coke samples shows the expected effect of the coarse porosity of this fuel. Figure 14 demonstrates the inadequacy of representing the specific surface of coals in terms of the size range of the fractions.

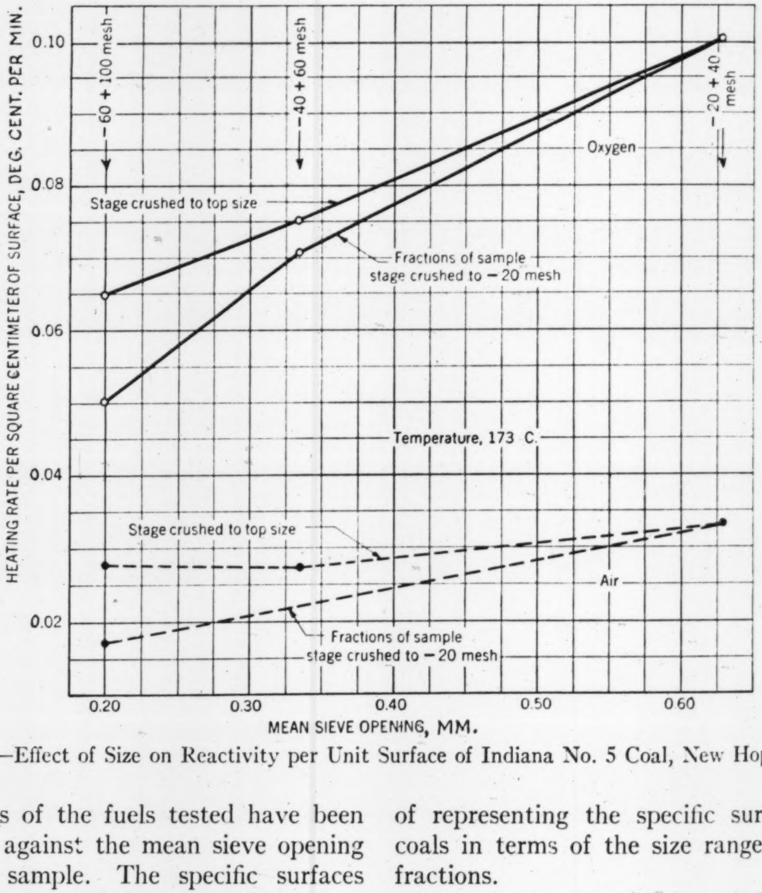


FIG. 17.—Effect of Size on Reactivity per Unit Surface of Indiana No. 5 Coal, New Hope Mine.

Variations in the nature of different sizes of a given fuel are best determined as differences in the reactivity per unit surface; that is, the rate of heating divided by the specific surface. Values of the heating rate per unit surface have been calculated for all the size fractions of the fuels tested in this investigation from the heating rate data of Fig. 10 to 13, and the specific surface data of Fig. 14, and have been plotted in Figs. 15, 16, and 17 against mean sieve opening of samples of Pittsburgh Bed coal, 900 C. experimental coke, and Indiana No. 5 Bed coal. Figure 15 shows that for the size fractions tested, stage-crushed Warden Mine coal has a maximum reactivity in the −40 +60 mesh size, and that the maximum variation between sizes is less than 10 per cent of the reactivity of the most reactive size. Therefore, for this fuel the −40 +60 mesh sample would be reasonably representative of the whole fuel. From Fig. 16 it is evident that essentially the same conclusions can be drawn for the 900 C. coke, except that the maximum reactivity occurs this time at the −20 +40 mesh size. In the case of the Indiana No. 5 Bed coal, Fig. 17, the results are entirely different. The heating rate per unit surface of the stage-crushed samples decreases progressively as the particle size decreases, the over-all change amounting to 35 per cent in the range of sizes studied. For the fractions of the sample stage-crushed to −20 mesh, the variation of reactivity with particle size is even greater, amounting to 50 per cent in the range of sizes studied. No single size is representative of this coal, and a knowledge of the size consist of a sample, rather than its specific surface, would be required for a reasonable estimation of heating rates.

TABLE IV.—PROXIMATE AND PETROGRAPHIC ANALYSES OF SIZED FRACTIONS OF INDIANA NO. 5 COAL, HIGH VOLATILE "C".

	Fractions of Stage Crushed −20 mesh			Stage Crushed to Top Size	
	−20 + 40 mesh Sample R126A	−40 +60 mesh Sample R126B	−60 +100 mesh Sample R126C	−40 +60 mesh Sample R126E	−60 +100 mesh Sample R126F
Per cent of total sample	63.5	18.6	4.7	47.0	39.6
Proximate analysis, moisture free, per cent:					
Volatile Matter	44.3	44.5	44.9	45.1	45.3
Fixed Carbon	45.1	44.6	44.2	43.8	44.0
Ash	10.6	10.9	10.9	11.1	10.7
Petrographic analysis, per cent:					
Anthraxylon[a]	22 } 72	31 } 71	28 } 72	24 } 72	25 } 77
Bright attritus[a]	50 }	40 }	44 }	48 }	52 }
Dull attritus[b]	25 } 28	25 } 29	23 } 28	27 } 28	21 } 23
Fusain[b]	3 }	4 }	5 }	1 }	2 }
Rate of heating per unit surface, in oxygen at 173 C., deg. C. per minute	0.102	0.071	.050	0.075	0.065

[a] Bright constituents.
[b] Dull constituents.

The exceptional behavior of the Indiana No. 5 Bed coal prompted a search for the responsible factors. Proximate and petrographic analyses were obtained of the various samples of this coal as shown in Table IV. There is little in these analyses to indicate the source of the variations in heating rate per unit surface found. With the exception of the higher percentage of bright constituents in sample R126F, compared to R126C, in line with the observed difference in reactivity of these samples, there is little correlation between analysis and reactivity. Apparently, factors not discernible by ordinary analytical methods have a pronounced effect on the reactivity of some coals. Further study of this aspect of the subject is indicated.

If in view of what is known of the nature of the materials, the assumption is made that the samples of Warden Mine coal and the 900 C. experimental coke have uniform properties independent of particle size, whereas the New Hope Mine coal does not, then it follows from these results that the reaction is essentially confined to the external surface of the material, as measured by the modified Lea-Nurse method, and the relative reactivities of two different materials of any particle size within the range of sizes studied here, may be taken as the ratio of the heating rates divided by the specific surfaces.

SUMMARY AND CONCLUSIONS

In order to provide a basis for correlation of reactivity with combustion and gasification studies, measurements were made of the reactivity of various solid fuels by the crossing-point method. Evidence accumulated to indicate that some of the basic assumptions of the method were not correct, and attention was therefore devoted to a re-examination of the method.

It was found first that the use of a thermocouple located outside of the reactivity tube introduced serious errors of calibration, yielded results which did not correspond to adiabatic heating at the crossing-point, and led to lack of reproducibility between different reactivity tubes. Relocation of the differential thermocouple inside the sample, 2 mm. radially distant from the sample thermocouple, following a study of the temperature distribution in the sample during heating, largely eliminated these difficulties. Thus, the calibration error of the differential thermocouple was reduced from +0.07 mv. to ±0.01 mv. The average deviation of the temperature at which the sample had a given heating rate was reduced to 0.7 C. for two different tubes, whereas, previously, systematic deviations between tubes as large as 8 C. were obtained. The adiabatic heating rates obtained with the new location of the thermocouples are 13.5 per cent higher than the corresponding value obtained with the previous arrangement of the thermocouples.

It was found, further, that the reaction with oxygen of the three fuels tested was of fractional order, 0.81 for a Pittsburgh Bed coal, 0.65 for an Indiana No. 5 Bed coal, and 0.71 for a 900 C. experimental coke. It is evident from these results that the procedure of calculating reactivity indices, t_{15} and t_{75}, from tests in oxygen and air for a limited range of heating rates and assuming a first order reaction, is not permissible.

The effect of the size consist of the sample on heating rate was also studied with −20 +40 mesh, −40 +60 mesh, and −60 +100 mesh, stage-crushed samples. The specific surface of each sample was determined by a modified Lea-Nurse air permeability method, and the reactivity per unit surface, the heating rate divided by the specific surface, calculated. The reactivity per unit surface varied little for the various size fractions of the Pittsburgh Bed coal, and the experimental coke. For the stage-crushed samples, Indiana No. 5 Bed coal, the reactivity per unit surface decreased rapidly as the average particle size decreased. This effect was even greater for non-stage-crushed samples of this coal. Thus, the reactivity per unit surface of the −60 +100 mesh fraction of a −20 mesh sample was only one-half of that of the −20 +40 mesh fraction. However, no significant variation was found in the proximate analyses of these samples, and little variation in the petrographic analyses, to account for the observed variations in reactivity.

The crossing-point method, as modified in this investigation, is a suitable means for measuring the adiabatic heating rates of small sizes of solid fuels. The range of heating rates conveniently measured is from 5 to 25 C. per min., covering a temperature range of about 50 C. The temperatures at which measurements are made will vary from about 150 C. for lignite to 500 C. for high-temperature coke. Measurements at four different heating rates in air and in oxygen may be made in one day.

The equipment required for this test is not too expensive, the major requirements being a good potentiometer and galvanometer. The thermocouple calibration equipment may be fabricated of glass or metal, and can be readily constructed in the laboratory. Thermocouple calibrations should be made at fairly frequent intervals because of the sensitivity of the method to errors of temperature measurement.

The results obtained by this method may be correlated with the composition of fuels and ultimately may provide a rational correlation with the behavior of fuels in practice, which is the purpose of any method that measures a fundamental property of fuels. Although applications will probably be mostly along research lines, considerable scope for the method may exist if the present trend toward specially prepared solid fuels continues. It is hoped that the availability of a suitable method for the determination of the reactivity of solid fuels will stimulate interest in such measurements and their application.

Acknowledgments:

A. C. Fieldner, Chief, Fuels and Explosives Division, and R. L. Brown, Chief, Coal Branch of the Bureau of Mines, authorized the investigation and made many helpful suggestions. The necessary fuel analyses were made under the direction of H. M. Cooper, Supervising Chemist, Coal-Analysis Section; and the petrographic analyses were made under the direction of W. A. Selvig, Supervising Chemist, Miscellaneous Analysis Section of the Bureau of Mines.

REFERENCES

(1) J. J. S. Sebastian and M. A. Mayers, "Coke Reactivity Determination by a Modified Ignition Point Method," *Industrial and Engineering Chemistry*, Vol. 29, pp. 118–124 (1937).

(2) R. A. Sherman, J. M. Pilcher, and H. N. Ostborg, "A Laboratory Test for the Ignitibility of Coal," ASTM BULLETIN, No. 112, October, 1941, p. 23.

(3) A. A. Orning, "Reactivity of Solid Fuels," *Industrial and Engineering Chemistry*, Vol. 36, pp. 814–816 (1944).

(4) A. A. Orning, S. Mallov, and M. Neff, "Reactivity of Solid Fuels to Air and Oxygen," *Industrial and Engineering Chemistry*, Vol. 40, pp. 429–432 (1948).

(5) E. F. Mueller and H. A. Burgess, "Standardization of the Sulphur Boiling Point," *Scientific Papers of the Bureau of Standards*, Vol. 15, pp. 163–184 (1919–1920).

(6) F. M. Lea and R. W. Nurse, "The Specific Surface of Fine Powders," *Journal*, Soc. Chem. Ind. *Transactions*, Vol. 58, pp. 277–83 (1939).

DISCUSSION

Mr. A. A. Orning[1] (*presented in written form*).—There can be no doubt that "the reactivity . . . defined as the rate of reaction of fuel and oxidizing gas, when determined under conditions such that it is a function of the properties of the fuel and not of the mass transport of reactant and product gases" is an important factor in combustion and gasification. However, the reactivity is only one factor, one which loses its importance at high temperatures and one which should be known for the fuel actually undergoing reaction. The contribution to the heat transfer system of heat release from those portions of a fuel bed for which the reactivity conditions apply is unknown, but probably of minor importance, except that reactivity indices can be used to indicate the relative temperature levels above which fuels will maintain ignition under different combustion conditions. The reactivity has more direct bearing upon "hold-fire" operation, but then must be known for the actual cokes involved rather than for the original fuels.

The authors have sought, with considerable effort, to obtain a more precise test without adding appreciably to either the basic concepts of the test or to its significance in relation to the use of fuels. They say that "Although the work reported . . . is restricted to two bituminous coals and one coke, the results obtained appear to have quite general application." It would be interesting to know on what basis such a general statement can be made.

An error of 14 C. in location of the crossing point as a result of an error of 1 C. in temperature difference compares favorably with a 10 C. error in reactivity index as previously reported.[2]

The care used in calibration of thermocouples and improving the homogeneity of thermocouple wire indicates the difficulty involved in determining the best criteria for adiabaticity. This is particularly true in an exothermic reaction with a positive temperature coefficient where hottest points tend to become hotter. It is difficult, in such a system, to justify the opinion that "The apparatus and procedure . . . give absolute values for the adiabatic rates of heating." Can we be certain that the location of thermocouples is correct for all temperature levels and differing reactivities? It hardly seems safe to generalize on the basis of a single sample.

It is interesting to find further confirmation of the failure of the first order law with respect to the partial pressure of oxygen. While one of three samples is found to deviate from Eq. 3, no alternative is offered. This equation was based upon a considerable number of samples,[3] but it was not possible to determine whether the scatter about the values predicted by the equation was due to experimental error or to inherent characteristics of the samples.

While the authors have contributed to the critical estimate of the test they offer no reason to modify the conclusion[3] that

---

[1] Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] A. A. Orning, *Industrial and Engineering Chemistry*, Vol. 36, pp. 813-816 (1944).

[3] A. A. Orning, S. Mallov, and N. Neff, Industrial and *Engineering Chemistry*, Vol. 40, pp. 429-432 (1948).

"The Coal Research Laboratory reactivity test, in either form, is quite useful as a research tool for studying the reactions between fuels and oxidizing gases, but it cannot be recommended as a commercial test unless it can be shown that more precise estimates of the reactivity indices are needed than can be obtained by . . . " estimates based on a knowledge of the proximate analysis of the fuel.

MESSRS. J. JONAKIN, P. COHEN, R. COREY, AND B. JAIN (*authors' closure*).—It will be readily acknowledged, as pointed out by Mr. Orning, that reactivity is but one of many factors affecting the combustion and gasification of solid fuels and that it probably loses its importance at high temperatures. It is also evident, however, that fuels are not directly introduced into combustion and gasification processes at high temperatures; thus, the low-temperature aspects of processes, for which reactivity is admittedly important, must be given serious consideration. For some processes, such as the heating of coal in storage, low rates of combustion as in fire places, and hold fire and pick-up characteristics in stokers, the application of reactivity data is directly indicated.

We have sought with considerable effort to obtain a more precise test because our experience has demonstrated the dangers of conclusions based on erroneous measurements. Measurements should be made as accurately as a reasonable expenditure of time and effort permits. The authors feel also that they have contributed to the basic concepts of the test, as demonstrated by the critical analysis given in the paper. As far as the significance of the test in relation to the use of fuels is concerned, the authors are well aware that this is the prime interest of fuel technologists, and have done the work described in this paper as a necessary prerequisite to the quantitative analyses of processes of combustion and gasification to determine the rôle therein of the reactivity of fuels.

Mr. Orning is not certain that the location of the thermocouples is correct for all temperature levels and differing reactivities, and bases his doubts on the exothermic nature and positive temperature coefficient of the reaction. It will be recalled that measurements in all cases will be confined to rates of heating between about 5 and 25 C. per minute, the conditions covered in the tests. Regardless of the reactivity of the fuel, which determines the temperature level at which measurements can be made, the same type of behavior found in these tests may with considerable certainty be expected to occur.

The error made in the location of the crossing point for a given error in the measurement of the differential temperature is a function of the nature of the material being tested, and the heating rate. For the particular test cited in the paper, the error in the crossing point would be 14 C. if an error of 1 C. was made in the measurement of the differential temperature, and this example was used to indicate the importance of accurate measurements of the differential temperature. As shown later in the paper, the error of measurement of the differential temperature was reduced to ±0.01 mv., or 1/4 C., which for the particular case cited would introduce an error of 3.5 C. in the crossing point. Since the error so made is always compensated by corresponding changes in the heating rate of the sample with change of the crossing point, the over-all error is even smaller. Thus, as cited in the paper, the average deviation of the experimental results determined in two different reactivity tubes, using thermocouples at the center and 2 mm. radially distant from the center, was only 0.7 C., when expressed as the difference between the experimentally determined temperature for a given heating rate, and the corresponding temperature of the best fitting Arrhenius curve.

In conclusion, it is repeated that this test is not recommended as a commercial test, nor can it properly be so recommended until careful work has demonstrated its utility. Correlation of fuel reactivity with proximate analyses is a valuable adjunct to experimental determinations but must be based on careful measurements using a firmly established experimental technique.

NONTOXIC RUBBER FOR GAS MASKS[*]

By S. H. Katz[1]

Synopsis

Some accelerators, antioxidants, and vulcanizing agents for natural rubber caused physiological reactions on the skin of wearers of gas masks. Limiting the chemical ingredients in the rubber to those proved by tests to cause no physiological effects and to exclude others was necessary. The patch tests employed in accordance with Chemical Corps specifications for acceptance of rubber parts of gas masks, which contact a wearer's skin, are outlined. Twenty-five accelerators and nineteen antioxidants which proved innocuous in the proportions needed, usually about 1 to 2 parts per 100 parts of rubber by weight, are listed. The listed materials would probably be innocuous if compounded with synthetic rubbers, but this has not been proved in the limited experience with synthetic rubber gas masks. The listed chemical materials should not be accepted as suitable in articles contacting a wearer's skin for extensive periods, other than gas masks, without first undergoing patch tests and other tests appropriate for the purposes.

Vulcanized natural rubber used in gas masks caused no physiological effects on the skin of wearers' faces until some varieties of antioxidants, accelerators, and vulcanizing-agent substitutes for elemental sulfur were introduced. The antioxidants multiplied the useful life of the gas mask rubber parts in storage or in service many times and eliminated the need of vulcanizing a surface covering of stockinette fabric to the rubber, a practice employed prior to the advent of modern compounding. In fact, the storage life of rubber parts of service types of gas masks in depots now exceeds their span of usefulness because the gas masks become obsolete and are replaced by newer standard types before the rubber becomes unserviceable. Moreover, when the masks are used in service the nonrubber parts usually deteriorate before the rubber rots. The rubber parts can then generally be salvaged and rebuilt into gas masks for additional service. The accelerators reduced the time required for vulvanizing rubber articles to a fraction of the time needed to vulcanize them without an accelerator. Production was accelerated and costs were decreased with faster vulcanization.

However, it has been necessary to limit the chemical ingredients to those proved by tests to cause no physiological effects when the gas masks are worn, and to exclude all others.

Synthetic rubber parts have also been used in gas masks and the same care to avoid physiological effects on the skin is needed as that required with natural rubber. Practical experience with the synthetic rubbers covers a relatively short period dating from the years of the recent war. Neoprene was employed in faceblanks and hose of the U. S. Army gas masks after the supplies of natural rubber tightened. Some properties of neoprene were less satisfactory than those of natural rubber, particularly the loss of flexibility after storage without heat in cold climates. Fortunately these gas masks were not permanently unserviceable because the flexibility could be restored by heating. The synthetic rubber compositions have appeared less prone to cause skin reactions. For instance butyl rubber could be compounded with tetramethyl thiuram disulfide, an excellent accelerator but toxic to the skin in natural rubber compounds **(1)**.[2] Tetramethyl thiuram disulfide proved innocuous in the amounts needed to compound butyl rubber for gas masks. The antioxidants and accelerators which proved nontoxic in the limited quantities added to natural rubber gas masks, usually less than two parts per hundred parts of rubber, are listed hereafter. It is probable that all or most of the antioxidants and accelerators in the lists would cause no toxic effects if employed in synthetic rubbers. But they should not be used in synthetic rubbers intended for prolonged contact with a person's skin without first undergoing careful tests in the prospective compounds to assure the absence of toxicity under the conditions of usage.

---

[*] Presented at the Fifty-first Annual Meeting of the Society, June 21-25, 1948.

[1] Chemical Corps Technical Command, Army Chemical Center, Maryland.

The limitations imposed on the choice of compounding ingredients have been applied only to faceblanks, nosecups, headpads, or other parts which contact the skin. The rubber in these particular parts is usually designated hereafter as "gas mask rubber"; this designation does not cover other rubber compositions in other parts such as inlet and outlet valves which do not contact the skin. "Natural gas mask rubber" or "synthetic gas mask rubber" designate the materials derived from the two indicated sources.

THE PATCH TEST

The "patch test," or "toxicity test," employed heretofore for acceptance of natural gas mask rubber parts, is outlined **(2)** in U. S. Army Specification No. 97-51-126, as follows:

> "*Toxicity test.*—The toxicity test shall be made with 10 test specimens, 1 inch square, cut from any one or several of the sample face blanks. The rubber specimens shall be applied beneath adhesive tape for 24 hours continuously to the under side of the upper arm of 10 persons selected in so far as possible for known sensitivity to rubber compounds. Any skin irritation due to physiological effects of the test specimens on any subject within 72 hours after the removal of the specimens shall be cause for rejection of the entire lot of faceblanks represented."

The ten subjects "selected in so far as possible for known sensitivity" were those who had shown reactions with rubber compositions previously, but these persons were not always available or were available in limited numbers. The number was increased to a maximum of 20 persons not necessarily selected, in the Chemical Corps Tentative Specification No. 197-51-271 for neoprene faceblanks **(3)**. This procedure required:

> "*Toxicity test.*—The toxicity test shall be made with 10 circular test specimens, 1 square inch in area cut from any one or several of the sample faceblanks. The toxicity test shall be made with every formulation of neoprene compound. The specimens shall be applied beneath adhesive tape for 24 hours continuously to the underside of the upper arm of ten persons. Any skin irritation due to physiological effects of the test specimen on any subject observed between 24 and 72 hours after the removal of the specimen shall be cause for repeating the test on another ten persons. Failure occurring in the second test shall be cause for rejection of the formulation. If more

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1298.

than one failure occurs in the first test, the formulation shall be rejected."

TABLE I.—RUBBER ACCELERATORS ACCEPTED FOR USE IN COMPOUNDS FOR FACEBLANKS, NOSECUPS OR OTHER PARTS OF U. S. ARMY GAS MASKS WHICH CONTACT A WEARER'S SKIN.

	Trade Names of Commercial Products Subjected to Patch Tests	Chemical Name or Description	Parts of Accelerator per 100 parts of Rubber by Weight
No. 1	A 7	Ethylidine aniline	1.18
No. 2	Accelerator 552	Piperidinium pentamethylene dithiocarbamate	1.0
No. 3	Accelerator 808	Condensation product of butyraldehyde and aniline	1.0
No. 4	Altax	Benzothiazyl disulfide	1.5
No. 5	APEF[a]	Zinc salt of mercaptobenzothiazole	0.6
No. 6	Aero AC 50	Reaction product of two mols of diorthotolyl guanidine and one mol of zinc chloride	1.0
No. 7	Captax	Mercaptobenzothiazole	1.5
No. 8	Delac A	Diphenyl guanidine acetate	1.25
No. 9	Delac O	Diphenyl guanidine oxalate	1.25
No. 10	Delac P[b]	Diphenyl guanidine phthalate	1.25
No. 11	DOTG	Diorthotolyl guanidine	1.0
No. 12	El Sixty	Dibenzothiazyl dimethyltolyl urea	1.0
No. 13	Guantal[b]	Diphenyl guanidine pthalate	0.3
No. 14	Latac	Hexamethylene-ammonium hexamethylene-dithiocarbamate	1.0
No. 15	2 MT	2-Mercaptothiazoline	1.0
No. 16	OXAF[a]	Zinc salt of mercaptobenzothiazole	0.65
No. 17	Phenex	Alpha-ethyl beta-propyl acryl aniline	1.24
No. 18	Pipsolene	Reaction product of carbon disulfide and methylene dipiperidine	0.70
No. 19	Quinoidine	A natural product	[d]
No. 20	Santocure	Condensation product of mercaptobenzothiazole and cyclohexylamine	1.0
No. 21	SPDX	Lead dithiocarbamate with a diphenyl ethylene diamine radical	1.24
No. 22	Tepidone	Sodium dibutyl-dithiocarbamate (50 per cent solution)	2.0[e]
No. 23	Trimene	An indefinite reaction product of ethyl chloride, formaldehyde and ammonia. This is mixed with stearic acid,1:1	3.1[f]
No. 24	Zenite[a]	Zinc salt of mercaptobenzothiazole (90 per cent) and paraffin wax 10 per cent	1.0
No. 25	Zenite A[ac]	Consists of 97 per cent Zenite and 3 per cent tetramethylthiurammonosulfide	1.0

[a] APEF, OXAF, and Zenite are chemically similar.
[b] Delac P and Guantal are chemically similar.
[c] Tetramethylthiurammonosulfide in any other combination is not permissible.
[d] No record of the amount is available.
[e] 2.0 parts of solution, equivalent to 1.0 part of solid.
[f] 3.1 parts equivalent to 1.55 parts of unmixed accelerator.

The information on the method of conducting the patch tests with natural or synthetic rubbers given in the specifications is very brief. Such tests however have been discussed in detail together with the medical aspects, by Schwartz and Tulipan in their textbook on occupational diseases of the skin (1). The medical phases are not considered in this paper except to mention that all the patch tests for acceptance of gas mask rubber parts conducted by the Chemical Corps were performed under the supervision of medical officers and the results were interpreted by them.

The rubber compositions accepted after patch tests on ten or more subjects with 24 hr. contact with the patches invariably yielded gas mask parts which in service caused no effects on the skin of the wearers. The tests provide a factor of safety because the gas masks are worn for periods not more than a few hours long. This factor proved ample, in prior experience with gas masks. But ten to twenty subjects and 24 hr. contact with the patches could be inadequate for rubber compositions intended for items of apparel or for therapeutic purposes if contact during actual use is longer than that of gas masks.

Notwithstanding the satisfactory results of prior experience with the specified patch tests the small numbers of subjects used for acceptance tests of gas mask rubber have been criticized as statistically unsound and an increase in the number of subjects may be required in future revisions of the specifications. Medical specialists have advocated three hundred subjects for a thorough test of a single composition. While a limited number of compositions might each be tested on hundreds of subjects, tests of this magnitude are obviously impractical for general use in acceptance procedures for gas mask rubber.

TABLE II.—RUBBER ANTIOXIDANTS ACCEPTED FOR USE IN COMPOUNDS FOR FACEBLANKS, NOSECUPS OR OTHER PARTS OF U. S. ARMY GAS MASKS WHICH CONTACT A WEARER'S SKIN.

	Trade Names of Commercial Products Subjected to Patch Tests.	Chemical Name or Description	Parts of Antioxidant per 100 parts of Rubber by Weight
No. 1	Agerite 41	Reaction product of diphenylamine and a hydrocarbon	1.39
No. 2	Agerite Resin	Aldol alphanapthylamine	2.0
No. 3	Agerite Resin D	Polymerized trimethyldihydroquinoline	1.39
No. 4	Agerite White	Symmetrical dibetanapthyl paraphenylenediamine	1.39
No. 5	Aminox Regular[a]	Reaction product of acetone and diphenylamine	1.0
No. 6	Aminox Special	Reaction product of acetone and diphenylamine	1.0
No. 7	Antox	Condensation product of butyraldehyde and aniline	1.0
No. 8	Betanox	Reaction product of acetone and phenylbetanaphthylamine	1.0
No. 9	BLE	Mixture of diphenylamine and substituted diphenylamines	2.9
No. 10	BLE Powder	Reaction product of acetone and diphenylamine (65 per cent) and N, N′-diphenyl paraphenylenediamine (35 per cent)	0.75
No. 11	Flectol H	2, 2, 4-trimethyl-1, 2-dihydroquinoline and polymers	1.0
No. 12	L-G-F	Para-(toluenesulfonamino) diphenylamine	1.36
No. 13	MUF	Para-(paratoluenesulfonamine) phenyl-paratolylamine	1.0
No. 14	Neozone A	Phenyl alphanaphthylamine	2.1
No. 15	Santoflex B	Reaction product of acetone and paraaminodiphenyl	1.0
No. 16	Santoflex BX	Mixture of Santoflex B and diphenylparaphenylenediamine	1.0
No. 17	Stabilite	Diphenyl ethylenediamine	1.24
No. 18	Stabilite Alba	Diorthotolyl ethylenediamine	1.24
No. 19	VGB	Reaction product of aniline and acetaldehyde	2.1

[a] Aminox Regular and Aminox Special differ in volatile materials, which were removed from the latter by vacuum treatment.

PERMISSIBLE ACCELERATORS AND ANTIOXIDANTS FOR NATURAL RUBBER

Table I gives the accelerators tested and approved for use in gas mask rubber. The table lists the trade names and the chemical names or descriptions of these materials. Table II gives similar information on antioxidants. The chemical names or designations are not rigidly precise in all cases, but with their trade names the materials are identified for the practical purposes of the rubber industry. The identifications were supplied by manufacturers of rubber products or manufacturers of the chemicals supplied to rubber manufacturers.

The patch tests were generally made with pieces cut from faceblanks submitted to the Chemical Corps for acceptance, but in some instances the patches were cut from test pieces having compositions intended for use in gas mask rubber. Some manufacturers of chemicals desiring to promote the use of their products for compounding gas mask rubber prepared and supplied the test pieces for approval and listing with the pertinent Chemical Corps specifications in order to inform the manufacturers who produce the required parts. The special test pieces were in most instances made to contain at least one part by weight of the chemical in question to 100 parts of rubber, and the specifications required at least 85 per cent rubber by volume prior to the advent of rubber shortages. If a chemical were needed in a proportion greater than one part per 100 parts of rubber, a rubber composition containing the recommended larger proportion was tested. The maximum proportions were 3.1 parts of Trimene, and in another instance 3 parts of tetraethyl thiuram disulfide per 100 of rubber in a composition containing no elemental sulfur, the sulfur being provided in chemical combination by the tetraethyl thiuram disulfide. The latter rubber composition proved toxic. The Trimene actually supplied only 1.55 parts of accelerator per 100 parts of rubber because it consisted of a mixture of equal weights of active accelerator and inactive stearic acid. BLE, an antioxidant, in the proportion of 2.9 parts per 100 of rubber constituted the largest proportion of actual antioxidant or accelerator found permissible. Four accelerators and one antioxidant were tested in amounts less than one part per 100 of rubber, and listed as permissible.

The lists of chemicals result from tests and experience extending from about 1930 when the toxic properties of some rubber compositions became evident. Investigations were then undertaken and the lists of acceptable materials were subsequently issued with the pertinent specifications. The lists gradually expanded as additional chemicals were tested and approved. Some of the items are no longer used extensively by the rubber industry but none have been deleted. While a maximum acceptable proportion of any chemical has not been determined, the listed chemicals may be considered usually safe in gas mask rubber when compounded in the proportions shown in Tables I and II. Retesting rubber compositions containing any chemical is occasionally practiced. A provision is quoted below from U. S. Army Specification 97–51–126:

> "This advance toxicity test . . . . shall not be considered as a final acceptance test for toxicity of the faceblanks as the United States Government reserves the right to conduct the toxicity test specified . . . . on all lots or shipments of faceblanks offered for acceptance regardless of any previous test that may have been conducted."

This provision cares for any circumstances where conceivably the influence of an ingredient of rubber other than the accelerator or the antioxidant may cause or may promote some physiological effects, or where some accelerator in combination with some particular antioxidant may produce effects even though they are separately innocuous. However such synergistic effects have not yet been evident.

TOXIC ACCELERATORS AND ANTIOXIDANTS

Rubber manufacturers and manufacturers of rubber chemicals cooperated with the Chemical Corps in the development of gas mask rubber with the understanding that trade confidences would be respected. Some materials proved toxic when tested in the formulations submitted. Their names are not available for publication in view of the cooperative status of the tests. Some manufacturers have held that disclosure of the names of chemicals not acceptable in some rubber parts of gas masks would curtail the use of those chemicals in other parts having no contact or effects on the skin, and might deter the use of the particular chemicals in other rubber goods where they present no hazard whatever. However tetramethyl thiuram monosulfide and tetraethyl thiuram disulfide were mentioned above as toxic in gas mask rubber faceblanks. Tetramethyl thiuram disulfide has been cited in the literature (4) as causing dermatitis on the hands of linemen wearing rubber gloves. Tetramethyl thiuram monosulfide is chemically very similar to tetraethyl thiuram disulfide and similar physiological effects could be expected. A number of chemicals which have caused dermatoses in the rubber industry and on users of some rubber products other than gas masks were cited by Schwartz and Tulipan (1). But chemicals hazardous in other circumstances may cause no effects on wearers of gas masks. For instance Schwartz and Tulipan (1) cite mercapto benzo thiazole as a cause of industrial dermatoses, but this accelerator proved innocuous in gas mask rubber.

CONCLUSIONS

1. Gas mask faceblanks, nosecups, and other parts worn against the skin should cause no physiological reactions on the skin.

2. Some accelerators, antioxidants, and other chemicals in gas mask rubber have caused reactions.

3. The patch test successfully screened the chemicals used or proposed for use in compounding gas mask rubber so as to permit the use of innocuous chemicals and to prevent the use of those which caused toxic effects.

4. The permissible accelerators and antioxidants approved to date for compounding natural rubber for gas mask parts contacting the skin are listed in Tables I and II.

5. The listed accelerators and antitoxidants would probably be nontoxic when compounded in synthetic rubbers for gas masks, but should not be employed in these rubbers without patch test proof that they are innocuous.

6. While the listed chemicals are suitable for compounding gas mask rubber when used in limited proportions, usually about 1 to 2 parts per 100 of rubber, they should not be accepted as suitable for use in rubber intended for other purposes without undergoing appropriate tests, including patch tests if the prospective use indicates the need of such testing.

REFERENCES

(1) L. Schwartz and L. Tulipan, "A Text-Book of Occupational Diseases of the Skin," Lea & Febiger, Philadelphia, p. 386 (also pp. 44 and 55) (1939).

(2) U. S. Army Specification No. 97-51-126, Faceblank Service (Fully Moulded) September 2, 1941.

(3) Amendment No. 4, November 25, 1944, on Chemical Corps Specification No. 197-51-271, Faceblank, Service, Assault (Fully Moulded) (Government Rubber-M), December 30, 1943.

(4) E. D. Osborne and E. D. Putnam, "Industrial Dermatoses with Special Reference to Allergy and Micotic Dermatoses," *Journal*, Am. Medical Assn., Vol. 99, 1932, pp. 972–977.

DISCUSSION

Mr. J. J. Allen[1] (*presented in written form*).—Mr. Katz has done a very fine piece of work in evaluating chemicals in natural and synthetic rubbers over the years. This paper represents very careful studies and experimental work.

The problem in determining the toxicity of rubber products and chemicals used in the rubber products is a difficult one, particularly in the selection of persons. Many people are immune to certain chemicals which will have a severe effect on others. It has been found that workers in rubber factories frequently are immune to accelerators and antioxidants which may prove to be toxic to other persons. Even office workers in rubber factories frequently develop an immunity. Therefore, as Mr. Katz points out, persons should be selected, if possible, for known sensitivity.

The conditions under which the tests are made also may have a bearing on the results. Tests made under conditions of normal or cool temperatures and in periods of low humidity may give entirely different results than under conditions of high humidity and temperature.

I believe, therefore, that anyone making rubber parts for use against the skin should compare results with the consumer so that both manufacturer and consumer are in agreement before starting any large scale production.

Mr. S. H. Katz (*author's closure*).—The medical officers of the Chemical Corps concerned with the testing of rubber compositions for toxic effects on the skin have recognized the possible differences in the sensitivity of the skin with differences in the climatic conditions of temperature and humidity, particularly for the extreme variations between winter and summer. Since control of the conditions was impracticable, the various tests were made whenever necessary under the existing climatic conditions in various months of the year. However, the tests with 24 hr. of contact between the rubber samples and the skin of subjects has given trustworthy evidence that gas mask rubber of the same composition may be employed safely, because no toxic effects on the skin of wearers have occurred when the compositions passing the tests during any one part of a year were worn in gas masks during any parts of subsequent years. The factors of safety provided by the shorter wearing periods for gas masks compared to the 24-hr. contact time in the patch tests probably compensates or overcompensates for any physiological differences in the skin under the various climatic conditions.

---

[1] Chief Chemist, Firestone Rubber and Latex Products Co., Fall River, Mass.

SUBJECT INDEX

VOLUME 48

A

Abrasion Test.

Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter—Seaman J. Tanenhaus and Gerald Winston. Published in A.S.T.M. BULLETIN, No. 154, October, 1948, p. 74 (TP232).

Acoustical Materials.

Report of Administrative Committee on Standards, Appendix II, Annual Report of the Board of Directors, 69.

Additives in Lubricants.

Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives. Appendix IX, Report of Committee D-2, 354.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Adhesives.

Adhesives. Report of Committee D-14, 445.

Some Factors Affecting Adhesive Bond Formation—H. P. Meissner and E. W. Merrill. Published in ASTM BULLETIN, No. 151, March, 1948, p. 80 (TP88).

Some Fundamentals of Glass Adhesion—Frank Moser. Published in A.S.T.M. BULLETIN, No. 150, January, 1948, p. 51 (TP23).

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers—Charles J. Seiler and A. D. McLaren. Published in ASTM BULLETIN, No. 155, December, 1948, p. 50 (TP258).

Administrative Committees.

See **Board of Directors.**

Aggregates.

Concrete and Concrete Aggregates. Report of Committee C-9, 263.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—W. H. Price and J. W. Robison, 962. Discussion, 968.

Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests—Delmar L. Bloem. Published in ASTM BULLETIN, No. 152, May, 1948, p. 76 (TP128). Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 81 (TP173).

Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete:

- Introduction, 1055.
- Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, 1057. Discussion, 1063.
- Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, 1067.
- Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Richard C. Mielenz and Leslie P. Witte, 1071. Discussion, 1104.
- A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and Theodore R. Smith, 1108. Discussion, 1115.
- Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, 1120. Discussion, 1126.

Research on Cobcrete Durability as Affected by Coarse Aggregate—Harold S. Sweet, 988. Discussion, 1017.

Aging.

Summary Report of Investigations of Aluminum-Base, Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.

Alkali Content.

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete:
- Introduction, 1055.
- Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, 1057. Discussion, 1063.
- Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, 1067.
- Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Richard C. Mielenz and Leslie P. Witte, 1071. Discussion, 1104.
- A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and Theodore R. Smith, 1108. Discussion, 1115.
- Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, 1120. Discussion, 1126.

Alloys, Non-Ferrous.

See also **Aluminum; Brass; Bronze; Copper; Magnesium; Zinc.**

Apparatus and Factors in Salt Fog Testing, V. M. Darsey and W. R. Cavanagh. Appendix, Report of Committee B-3, 153.

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys—R. B. Smith. Published in ASTM BULLETIN, No. 151, March, 1948, p. 50 (TP58).

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic B Ending—Harry Majors, Jr. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).

Chemical Analysis of Metals. Report of Committee E-3, 505

Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 178.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Die-Cast Metals and Alloys. Report of Committee B-6, 187.

The Effect of Speed of Testing on Magnesium-Base Alloys—Symposium on Speed of Testing—A. A. Moore, 1133.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.

Electrical-Heating, Resistance, and Related Alloys. Report of Committee B-4, 176.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—P. G. Jones and W. J. Worley, 648. Discussion 663.

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form.—H. L. Burghoff and A. I. Blank, 709. Discussion, 733.

Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 198.

Metallography. Report of Committee E-4, 510.

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Panel Discussion on Corrosion of Pressure Vessels:
- Station Design and Material Composition as Factors in Boiler Corrosion—R. B. Donworth, 897. Discussion, 905.
- Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, 907. Discussion, 926.

A Review of the Tin Situation, Marshall W. Tuthill. Appendix, Report of Committee B-2, 146.

Recommendations Affecting Standards for Copper and Copper Alloys, Cast and Wrought. Appendix, Report of Committee B-5, 181.

Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion, 754.

What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Wires for Electrical Conductors. Report of Committee B-1, 138.

Alloy Steel.

See also **Steel.**
Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, 610.
Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, 591. Discussion, 608.
Filler Metal. Report of Joint Committee, 210.
High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, 555. Discussion, 589.
Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Report of Committee A-10, 134.
Magnetic Properties. Report of Committee A-6, 125.
Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, 664. Discussion, 690.

Aluminum.

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys.—R. B. Smith. Published in ASTM BULLETIN, No. 151, March, 1948, p. 50 (TP58).
Chemical Analysis of Metals. Report of Committee E-3, 505.
Die-Cast Metals and Alloys. Report of Committee B-6, 187.
An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—P. G. Jones and W. J. Worley, 648. Discussion, 663.
Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.
Filler Metal. Report of Joint Committee, 210.
Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 198.
Metals and Plastics—Robert G. Chollar. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP132); Discussion, p. 87 (TP139).
Summary Report of Investigations of Aluminum-Base, Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.
What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).
Wires for Electrical Conductors. Report of Committee B-1, 138.

Amaloy Coatings.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Annapolis Exposure Tests.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.

Annual Meeting.

Annual Report of the Board of Directors, 35.
Summary of Proceedings of the Fifty-first Annual Meeting, 1.

Anodic Coatings.

Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 198.

Antifreezes.

See **Engine Antifreezes.**

Antioxidants.

Nontoxic Rubber for Gas Masks—S. H. Katz, 1293. Discussion, 1299.

Appearance Tests.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Asbestos.

Textile Materials. Report of Committee D-13, 434.

Asbestos Cement Products.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Asphalt.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.

Road and Paving Materials. Report of Committee D-4, 383.

Atmospheric Corrosion.

Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, 610.

Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, 591. Discussion, 608.

Galvanic and Electrolytic Corrosion. Report of Subcommittee VIII, Committee B-3, 167.

Atomic Energy.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture—Paul C. Aebersold, 527.

Automotive Equipment.

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Petroleum Products as Related to Automotive Equipment, 1216.

Aviation Gasoline.

See also **Gasoline.**

Better Lead Scavenging Needed for Aviation Gasoline—E. A. Droegemueller. Published in ASTM BULLETIN, No. 154, October, 1948, p. 53 (TP211).

B

Bacteria in Water.

Proposed Method for Identification of Types of Microorganisms in Industrial Waters. Appendix, Report of Committee D-19, 486.

Ball-Bearing Greases.

Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.

Barbed Wire.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Bast and Leaf Fibers.

Textile Materials. Report of Committee D-13, 434.

Bearing Metal.

Metal Powders and Metal Powder Products. Report of Committee B-9, 208.

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Report of Administrative Committee on Simulated Service Testing. Appendix VI, Annual Report of the Board of Directors, 86.

Benzene.

Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry. Appendix XII, Report of Committee D-2, 374.

Bird Film Applicator.

Mechanical Operation of the Bird Film Applicator—A. E. Jacobsen and H. S. Jensen. Published in ASTM BULLETIN, No. 151, March, 1948, p. 95 (TP103).

The Preparation of Uniform Films and Their Dependence on the Irregularities of the Base Surface—Stig Johanson. Published in ASTM BULLETIN, No. 155, December, 1948, p. 53 (TP261).

Bituminous Materials.

Road and Paving Materials. Report of Committee D-4, 383.

Summary of Softening Point and Melting Point Methods Used for Testing Rosins and Resins, Report of Subcommittee I of Committee D-17, 457.

Board of Directors.

Annual Report of the Board of Directors, 35.

Report of Auditors, 58.

Report of Administrative Committee on Standards, 69

Report of Administrative Committee on Research, 73.

Board of Directors—*continued.*

Report of Administrative Committee on Papers and Publications, 76.
Report of Administrative Committee on Ultimate Consumer Goods, 84.
Report of Administrative Committee on Simulated Service Testing, 86.
Report of Administrative Committee on District Activities, 88.

Boiler Feedwater.

Panel Discussion on Corrosion of Pressure Vessels:
  Station Design and Material Composition as Factors in Boiler Corrosion—R. B. Donworth, 897. Discussion, 905.
  Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, 907. Discussion, 926.
Industrial Water. Report of Committee D-19, 480.

Bolting Materials.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.
Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.
High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.

Bond Strength.

The Effect of Repeated Loading on the Bond Strength of Concrete—C. W. Muhlenbruch, 977. Discussion, 986.

Boxes.

Paper and Paper Products. Report of Committee D-6, 395.
Shipping Containers. Report of Committee D-10, 415.

Brass.

Chemical Analysis of Metals. Report of Committee E-3, 505.
Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 178.
Die-Cast Metals and Alloys. Report of Committee B-6, 187.
Recommendations Affecting Standards for Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, Appendix, 181.

Brick.

Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, 1020. Discussion, 1040.
Refractories. Report of Committee C-8, 256.

Brittleness Test.

An Improved Low-Temperature Brittleness Test—E. F. Smith and G. J. Dienes. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204); Discussion, p. 49 (TP207).

Bronze.

See also **Copper.**
Chemical Analysis of Metals. Report of Committee E-3, 505.
Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 178.
Recommendations Affecting Standards for Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, Appendix, 181.

Building Materials.

Fire Tests of Materials and Construction. Report of Committee E-5, 512.
Methods of Testing Building Constructions. Report of Committee E-6, 515.
Natural Building Stones. Report of Committee C-18, 275.

Butadiene.

Proposed Method of Test for Butadiene Dimer in Polymerization Grade Butadiene. Appendix III, Report of Committee D-2, 312.
Proposed Method of Test for Nonvolatile Residue of Polymerization Grade Butadiene. Appendix IV, Report of Committee D-2, 316.
Proposed Method of Test for Separation of Residue from Butadiene. Appendix II, Report of Committee D-2, 309.

C

Cable.

Wires for Electrical Conductors. Report of Committee B-1, 138.

Cadmium Coatings.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Calibration, Methods of.

Dynamic Calibration Method Uses Modified Proving Ring—R. C. A. Thurston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 50 (TP208).

A Method of Calibrating Extensometers—W. C. Aber and F. M. Howell. Published in ASTM BULLETIN, No. 155, December, 1948, p. 33 (TP241); Discussion, p. 35 (TP 243).

Calorimeter.

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Calorized Coatings.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Carloading Methods.

The Use of Test Track in Developing Good Packaging and Carloading Methods—A. N. Perry. Published in ASTM BULLETIN, No. 155, December, 1948, p. 38 (TP246).

Castings.

Radiographic Testing. Report of Committee E-7, 517.

Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Cast Iron.

Cast Iron. Report of Committee A-3, 102.

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, 841. Discussion, 853.

Fatigue and Static Load Tests of an Austenitic Cast Iron at Elevated Temperatures—W. Leighton Collins, 696. Discussion, 706.

Cathode Nickel.

Abstracts of Papers on Cathode Nickel. Published in ASTM BULLETIN, No. 152, May, 1948:

A Standard Diode for Radio Tube Cathode—Core-Material Approval Tests, by Robert L. McCormack, p. 37.

Testing Cathode Materials in Factory Production, by J. T. Acker, p. 38.

European Practice in the Manufacture of Cathodes, by T. H. Briggs, p. 39.

Cement.

Cement. Report of Committee C-1, 244.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—W. H. Price and J. W. Robison, 962. Discussion, 968.

Magnesium Oxychloride Cements. Report of Committee C-2, 253.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements—Harry E. Chisholm. Published in ASTM BULLETIN, No. 154, October, 1948, p. 37 (TP195); Discussion, p. 39 (TP 197).

Proposed Method of Test for Setting Time of Hydraulic Cement in Mortar. Appendix, Report of Committee C-1, 250.

Recent Development in Portland Cement (With a Thumbnail Sketch of the Industry) (Condensed Version)—J. F. Barton. Published in ASTM BULLETIN, No. 150, January, 1948, p. 18.

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete:

Introduction, 1055.

Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, 1057. Discussion, 1063.

Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, 1067.

Cement—*continued.*

Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Richard C. Mielenz and Leslie P. Witte, 1071. Discussion, 1104.

A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and Theodore R. Smith, 1108. Discussion, 1115.

Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, 1120. Discussion, 1126.

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., 944. Discussion, 959.

Thermal Insulating Materials. Report of Committee C-16, 271.

Chain-Link Fence.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Chemical Analysis.

Abstract of Papers on Organic Reagents for Metal Analysis. Published in ASTM Bulletin, No. 151, March, 1948:

Organic Reagents for Gravimetric Analysis—John F. Flagg, p. 31.

The Use of Organic Solvents in Metal Analysis—C. J. Rodden, p. 31.

Organic Reagents in Colorimetric Analysis—John H. Yoe, p. 32.

Organic Reagents for Volumetric Analysis—John F. Flagg, p. 32.

Oxidation-Reduction Indicators—G. Frederick Smith, p. 33.

Cement. Report of Committee C-1, 244.

Chemical Analysis of Metals. Report of Committee E-3, 505.

Spectrographic Analysis. Report of Committee E-2, 503.

Chemical Reactivity.

See **Reactive Materials in Concrete.**

Chromic Acid.

The Action of "Chromic Acid" on Zinc Coatings—D. J. Swaine. Published in ASTM BULLETIN, No. 154, October, 1948, p. 52 (TP 210). Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 49 (TP11).

Chromium.

Changes Found in Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, 841. Discussion, 853.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

Clay.

Clay Pipe. Report of Committee C-4, 254.

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., 944. Discussion, 959.

Coal.

Coal and Coke. Report of Committee D-5, 389.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method—J. Jonakin, P. Cohen, R. Corey, and B. Jain, 1269. Discussion, 1290.

Coatings.

See **Electrodeposited Coatings; Paint.**

Code Systems.

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys—R. B. Smith. Published in ASTM BULLETIN, No. 151, March, 1948, p. 50 (TP58).

Coke.

Coal and Coke. Report of Committee D-5, 389.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method—J. Jonakin, P. Cohen, R. Corey, and B. Jain, 1269. Discussion. 1290.

Color Metallography.

Summary of Proceedings of the Symposium on Metallography in Color, 942.

Color Index.

Photoelectric Measurement of a Color Index of Liquid Petroleum Products—Louis Lykken. Published in ASTM BULLETIN, No. 154, October, 1948, p. 68 (TP226).

Compression Testing.

The Effect of Speed of Testing on Magnesium-Base Alloys—Symposium on Speed of Testing—A. A. Moore, 1133.

Concrete.

Concrete and Concrete Aggregates. Report of Committee C-9, 263.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—W. H. Price and J. W. Robison, 962. Discussion, 968.

The Effect of Repeated Loading on the Bond Strength on Concrete—C. W. Muhlenbruch, 977. Discussion, 986.

Research on Concrete Durability as Affected by Coarse Aggregate—Harold S. Sweet, 988. Discussion, 1017.

Seventh Sanford E. Thompson Award. Summary of Proceedings of the Fifty-first Annual Meeting, 22. Report of Committee C-9, 263.

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete:

- A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and Theodore R. Smith, 1108. Discussion, 1115.
- Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, 1067.
- Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, 1057. Discussion, 1063.
- Introduction, 1055.
- Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, 1120. Discussion, 1126.
- Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Richard C. Mielenz and Leslie P. Witte, 1071. Discussion, 1104.

Conditioning.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Electrical Insulating Materials. Report of Committee D-9, 402.

Methods of Testing. Report of Committee E-1, 498.

Report of Administrative Committee on Simulated Service Testing. Appendix VI, Annual Report of the Board of Directors, 86.

Consistency.

The Light-Weight Cone for Penetration of Soft Greases—Prepared by L. C. Brunstrum. Published in ASTM BULLETIN, No. 154, October, 1948, p. 66 (TP224).

Methods of Testing. Report of Committee E-1, 498.

A Worker-Consistometer for Lubricating Greases—S. A. McKee and H. S. White. Published in ASTM BULLETIN, No. 153, August, 1948, p. 90 (TP182); Discussion, p. 97 (TP189).

Consumer Goods.

Report of Administrative Committee on Ultimate Consumer Goods. Appendix V, Annual Report of the Board of Directors, 84.

Contact Metals.

Electrical-Heating, Resistance and Related Alloys. Report of Committee B-4, p. 176.

Containers.

Paper and Paper Products. Report of Committee D-6, 395.

Shipping Containers. Report of Committee D-10, 415.

Copper.

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending—Harry Majors, Jr. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).

Chemical Analysis of Metals. Report of Committee E-3, p. 505.

Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 178.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form.—H. L. Burghoff and A. I. Blank, 709. Discussion, 733.

Copper—*continued.*

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Quantitative Evaluation of Corrosive Conditions—W. F. Bonwitt and I. Eisen. Published in ASTM BULLETIN, No. 151, March, 1948, p. 84 (TP92); Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 44 (TP 202).

Recommendations Affecting Standards for Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, Appendix, 181.

What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Copper-Bearing Steel.

See also **Steel.**

Corrosion of Iron and Steel. Report of Committee A-5, 104.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee, A-5, 114.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.

Copper Covered Wires.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Copper Wire.

Wires for Electrical Conductors. Report of Committee B-1, 138.

Core Loss.

Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Corrosion.

See also **Weathering.**

The Action of "Chromic Acid" on Zinc Coatings—D. J. Swaine. Published in ASTM BULLETIN, No. 154, October, 1948, p. 52 (TP210). Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 49 (TP11).

Apparatus and Factors in Salt Fog Testing, V. M. Darsey and W. R. Cavanagh. Appendix, Report of Committee B-3, 153.

Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, 610.

Corrosion of Iron and Steel. Report of Committee A-5, 104.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab, 1201. Discussion, 1211.

Die-Cast Metals and Alloys. Report of Committee B-6, 187.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

Engine Antifreezes. Report of Committee D-15, 448.

Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, 591. Discussion, 608.

Field Tests and Inspections of Bare, and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Galvanic and Electrolytic Corrosion. Report of Subcommittee VIII, Committee B-3, 167.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.

Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Report of Committee A-10, 134.

An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster, 628. Discussion, 645.

Laboratory Corrosion Tests of Iron and Steel Pipes—G. A. Ellinger, L. J. Waldron, and S. B. Marzolf, 618.

Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 198.

Panel Discussion on Corrosion of Pressure Vessels:
  Station Design and Material Composition as Factors in Boiler Corrosion—R. B. Donworth, 897. Discussion, 905.
  Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, 907. Discussion, 926.
Quantitative Evaluation of Corrosive Conditions—W. F. Bonwitt and I. Eisen. Published in ASTM BULLETIN, No. 151, March, 1948, p. 84 (TP92); Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 44 (TP202).
Requirements of an Engine Antifreeze and Methods of Evaluation—D. H. Green, J. C. Kratzer, P. I. Emch. Published in ASTM BULLETIN, No. 154, October, 1948, p. 57 (TP215); Discussion, p. 65 (TP 223).
Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.
A Visual Rating System for Rusted Steel Specimens—Harry L. Faigen. Published in ASTM BULLETIN, No. 154, October, 1948, p. 39 (TP197).

Corrugated Paper.
Load Concentration in Corrugated Paper—J. H. Toulouse and P. J. Barcus. Published in ASTM BULLETIN, No. 152, May, 1948, p. 77 (TP129).

Corrosion-Resisting Steels.
See also **Alloy Steels, Corrosion.**
Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.
Galvanic and Electrolytic Corrosion. Report of Subcommittee VIII, Committee B-3, 167.
Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Report of Committee A-10, 134.

Cotton.
See also **Textile Materials.**
Textile Materials. Report of Committee D-13, 434.

CR-39
Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Crossarms.
Wood. Report of Committee D-7, 400.

Cutting Oils.
Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.
Symposium on Cutting Oils. Published in ASTM BULLETIN, No. 151, March, 1948:
  Water-Mixed Cutting Fluids—William H. Oldacre, p. 45 (TP53).
  Cutting Fluids—E. H. Reehl, p. 47 (TP55).
  Correlation of Laboratory Tests of Cutting Fluids with Field Tests—D. J. Wangelin, p. 48 (TP56).

Creep Testing.
High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.
High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Varibles in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, 555. Discussion, 589.
The Importance of the Time Factor in the Study of Textile Properties—Edward R. Schwarz. Published in ASTM Standards on Textile Materials, October, 1948, p. 485.
Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, 815. Discussion, 825.
The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.
Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion, 754.
The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation

Creep Testing—*continued.*

Energy Theory—W. N. Findley, C. H. Adams, and W. J. Worley, 1217.

Cutting Oils.

Symposium on Cutting Oils. Published in ASTM BULLETIN, No. 151, March, 1948:
 Water-Mixed Cutting Fluids—William H. Oldacre, p. 45 (TP53).
 Cutting Fluids—E. H. Reehl, p. 47 (TP55).
 Correlation of Laboratory Testing of Cutting Fluids with Field Tests—D. J. Wangelin, p. 48 (TP56).

D

Dams.

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams—W. G. Holtz, 1240.

Data, Presentation of.

See **Statistical Analysis.**

Definitions.

Nomenclature and Definitions. Report of Committee E-8, 519.

Deformation of Metals.

Report of Administrative Committee on Simulated Service Testing. Appendix VI, Annual Report of the Board of Directors, 86.

Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Density.

An Instrument for Determining the Volume and Bulk Density of Granular Materials—William E. Gross and John C. Goshorn. Published in ASTM BULLETIN, No. 151, March, 1948, p. 55 (TP63).

Detonation Meter.

Optional Use of Detonation Meter for Motor and Research Methods. Appendix VI, Report of Committee D-2, 328.

Summary Report on Investigation of the Detonation Meter, Model 501. Appendix VIII, Report of Committee D-2, 340.

Die Castings.

Die-Cast Metals and Alloys. Report of Committee B-6, 187.

Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.

Diesel Fuel Oil.

Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Dipentene.

Naval Stores. Report of Committee D-17, 451.

Distillation Test.

Proposed Method of Test for Butadiene Dimer in Polymerization Grade Butadiene. Appendix III, Report of Committee D-2, 312.

Proposed Method of Test for Separation of Residue from Butadiene. Appendix II, Report of Committee D-2, 309.

District Activities.

Report of Administrative Committee on District Activities. Appendix VII, Annual Report of the Board of Directors, 88.

Ductility.

Summary of the Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Dynamic Calibration.

Dynamic Calibration Method Uses Modified Proving Ring—R. C. A. Thurston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 50 (TP208).

E

Elastomers.

An Improved Low-Temperature Brittleness Test—E. F. Smith and G. J. Dienes. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204); Discussion, p. 49 (TP207).

Electric Strain Gage.

Measurement and Elimination of Inertia Effects in Textile Testing Equipment by Means of Electric Strain Gages—Herbert F. Schiefer. Published in A.S.T.M. BULLETIN, No. 150, January, 1948, p. 43 (TP15).

Electrical Insulating Materials.

Electrical Insulating Materials. Report of Committee D-9, 402.
Electrical Properties of Plastics—A. J. Warner. Published in ASTM BULLETIN, No. 153, August, 1948, p. 60 (TP152).
Plastics. Report of Committee D-20, 490.
Recommendations Affecting Standards for Electrical Insulating Materials. Appendix, Report of Committee D-9, 410.

Electrical Heating and Resistance Alloys.

Electrical-Heating, Resistance, and Related Alloys. Report of Committee B-4, 176.

Electrodeposited Coatings.

Corrosion of Iron and Steel. Report of Committee A-5, 104.
Electrodeposited Metallic Coatings. Report of Committee B-8, 206.
Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Electrolytic Corrosion.

See also **Corrosion.**
Galvanic and Electrolytic Corrosion. Report of Subcommittee VIII, Committee B-3, 167.
Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Engine Antifreezes.

Engine Antifreezes. Report of Committee D-15, 448.
Requirements of an Engine Antifreeze and Methods of Evaluation—D. H. Green, J. C. Kratzer, and P. I. Emch. Published in ASTM BULLETIN, No. 154, October, 1948, p. 57 (TP215); Discussion, p. 65 (TP223).

Estron.

Textile Materials. Report of Committee D-13, 434.

Extensometers.

Method of Calibrating Extensometers—W. C. Aber and F. M. Howell. Published in ASTM BULLETIN, No. 155, December, 1948, p. 33 (TP241); Discussion, p. 35 (TP243).

F

Fabrication.

Methods of Testing Building Constructions. Report of Committee E-6, 515.

Fabrics.

See **Textile Materials.**

Fatigue Testing.

Dynamic Calibration Method Uses Modified Proving Ring—R. C. A. Thurston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 50 (TP208).
Effect of Fatigue on Tension-Impact Resistance—William H. Hoppmann II. Published in ASTM BULLETIN, No. 155, December, 1948, p. 36 (TP244); Discussion, p. 37 (TP245).
Fatigue Properties of Some Coppers and Copper Alloys in Strip Form—H. L. Burghoff and A. I. Blank, 709. Discussion, 733.
Fatigue. Report of Committee E-9, 522.
Fatigue and Static Load Tests of An Austenitic Cast Iron at Elevated Temperatures—W. Leighton Collins, 696. Discussion, 706.
An Hypothesis for the Determination of Cumulative Damage in Fatigue—F. E. Richart, Jr., and N. M. Newmark, 767. Discussion 799.
An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster, 628. Discussion, 645.
Report of Administrative Committee on Simulated Service Testing. Appendix VI, Annual Report of Board of Directors, 86.
Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, 664. Discussion, 690.

Fatigue Testing—*continued.*

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above The Endurance Limit—James B. Duke, 755. Discussion, 763.

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion, 754.

Fence.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Ferro-Alloys.

Ferro-Alloys. Report of Committee A-9, 133.

Fiberboard.

Paper and Paper Products. Report of Committee D-6, 395.

Fibers.

Textile Materials. Report of Committee D-13, 434.

Filler Metal.

Filler Metal. Report of Joint Committee, 210.

Finances.

Report of the Auditors for the Fiscal Year, January 1 to December 31, 1947. Appendix I, Annual Report of the Board of Directors, 58.

Firebrick.

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Fire Tests.

Fire Tests of Materials and Construction. Report of Committee E-5, 512.

Flammability.

Symposium on Flammability. Published in ASTM BULLETIN, No. 150, January, 1948:

Flammability of Textiles for Garment Purposes—Frederic Bonnet, p. 29 (TP1).

Flameproofing in Theory and Practice—R. W. Little, p. 34 (TP6).

The Retailer's Viewpoint on the Flammability of Textiles—Charles W. Dorn, p. 36 (TP8).

Flow.

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C.—F. E. Piech and W. E. Gloor. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Forming Properties.

Summary of the Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Freezing-and-Thawing Tests.

Research on Concrete Durability as Affected by Coarse Aggregate—Harold S. Sweet, 988. Discussion, 1017.

Fuel Oil.

Summary of Proceedings of the Forum on Tractor Fuels, 1215.

Optional Use of Detonation Meter for Motor and Research Methods. Appendix VI, Report of Committee D-2, 328.

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases. Appendix V, Report of Committee D-2, 318.

Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Fungus.

Fungus Resistance of Treated Vegetable Fiber Gaskets—Sigmund Berk. Published in ASTM BULLETIN, No. 150, January, 1948, p. 46 (TP18).

The Effect of Fungi and Humidity on Plastics—John Leutritz, Jr. Published in ASTM BULLETIN, No. 152, May, 1948, p. 88 (TP140).

G

Galvanic Corrosion.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Galvanic and Electrolytic Corrosion. Report of Subcommittee VIII, Committee B-3, 167.

Galvanized Sheet and Wire.

The Action of "Chromic Acid" on Zinc Coatings—D. J. Swaine. Published in ASTM BULLETIN, No. 154, October, 1948, p. 52 (TP210). Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 49 (TP11).

Gas Mask Materials.

An Instrument for Determining the Volume and Bulk Density of Granular Materials—William E. Gross and John C. Goshorn. Published in ASTM BULLETIN, No. 151, March, 1948, p. 55 (TP63).

Nontoxic Rubber for Gas Masks—S. H. Katz, 1293. Discussion, 1299.

Gaskets.

Fungus Resistance of Treated Vegetable Fiber Gaskets—Sigmund Berk. Published in ASTM BULLETIN, No. 150, January, 1948, p. 46 (TP18).

Gaseous Fuels.

Gaseous Fuels. Report of Committee D-3, 381.

Gasoline.

Better Lead Scavenging Needed for Aviation Gasoline—E. A. Droegemueller. Published in ASTM BULLETIN, No. 153, October, 1948, p. 53 (TP211).

Induction Period Calculator—E. L. Korb. Published in ASTM BULLETIN, No. 153, August, 1948, p. 99 (TP191).

Petroleum Products and Lubricants. Report of Committee D-2, 287. Supplementary Report, 298.

Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry. Appendix XII, Report of Committee D-2, 374.

Summary of Meeting on Petroleum Product as Related to Automotive Equipment, 1216.

Summary of Proceedings of the Forum on Tractor Fuels, 1215.

Summary Report on Investigation of the Detonation Meter, Model 501. Appendix VIII, Report of Committee D-2, 340.

Gas Turbine Material.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, 555. Discussion, 589.

Gehman Torsional Apparatus.

Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.

Glass.

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Glass and Glass Products. Report of Committee C-14, 270.

The Preparation of Uniform Films and Their Dependence on the Irregularities of the Base Surface—Stig Johanson. Published in ASTM BULLETIN, No. 155, December, 1948, p. 53 (TP261).

Some Fundamentals of Glass Adhesion—Frank Moser. Published in ASTM BULLETIN, No. 150, January, 1948, p. 51 (TP23).

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Glass Textiles.

See also **Textile Materials.**

Textile Materials. Report of Committee D-13, 434.

Gloss.

The Gloss Measurement of Paint Finishes—Richard S. Hunter. Published in ASTM BULLETIN, No. 150, January, 1948, p. 72 (TP44).

Glues.

See also **Adhesives.**
Adhesives. Report of Committee D-14, 445.

Grain Size.

Metallography. Report of Committee E-4, 510.

Graphitization.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.
Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Grease.

The Light-Weight Cone for Penetration of Soft Greases—Prepared by L. C. Brunstrum. Published in ASTM BULLETIN, No. 154, October, 1948, p. 66 (TP224).
Petroleum Products and Lubricants. Report of Committee D-2, 287. Supplementary Report, 298.
Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.
Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease. Appendix I, Report of Committee D-2, 303.
The Thixotropic Characteristics of Lubricating Oil Greases—L. W. McLennan and G. H. Smith. Published in ASTM BULLETIN, No. 152, May, 1948, p. 71 (TP123).
Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.
A Worker-Consistometer for Lubricating Greases—S. A. McKee and H. S. White. Published in ASTM BULLETIN, No. 153, August, 1948, p. 90 (TP182); Discussion, p. 97 (TP189).

Gypsum.

Gypsum. Report of Committee C-11, 267.

H

Hardness Testing.

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending—Harry Majors, Jr. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).
A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range—Richard C. Weatherwax, E. C. O. Erickson and Alfred J. Stamm. Published in ASTM BULLETIN, No. 153, August, 1948, p. 84 (TP176).

Hardware.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Heat Determination.

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Heat Distortion.

A New Interpretation of Heat Distortion Data of Plastics—L. S. Moody and K. B. Goldblum. Published in ASTM BULLETIN, No. 150, January, 1948, p. 55 (TP27).

Hiding Power.

A Method for the Routine Determination of Dry Hiding Power of White Paints—F. C. Schmutz and J. M. Gallagher. Published in ASTM BULLETIN, No. 153, August, 1948, p. 77 (TP169).

High-Speed Radiography.

Frankford Arsenal Experience with High-Speed Radiography—E. R. Thilo. Published in ASTM BULLETIN, No. 150, January, 1948, p. 69 (TP41).
One-Millionth Second Radiography and Its Development—Charles M. Slack and Louis F. Ehrke. Published in ASTM BULLETIN, No. 150, January, 1948, p. 59 (TP31).

High-Temperature Materials.

See also **Refractories; Temperature, Effect of.**

Effect of Temperature on the Properties of Metals. Report of Joint Committee 212.

High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.

Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Humidity.

See also **Conditioning.**

The Effect of Fungi and Humidity on Plastics —John Leutritz, Jr. Published in ASTM BULLETIN, No. 152, May, 1948, p. 88 (TP140).

I

Ignition Temperatures.

Discussion of Paper on the Ignition Temperature of Rigid Plastics. Published in ASTM BULLETIN, No. 151, March, 1948, p. 66 (TP74).

Immersion Testing.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Impact Testing.

Discussion of Paper on the Resistance to Impact Loading of Plastics. Published in ASTM BULLETIN, No. 153, August, 1948, p. 59 (TP151); No. 154, October, 1948, p. 45 (TP203).

Effect of Fatigue on Tension-Impact Resistance—William H. Hoppmann II. Published in ASTM BULLETIN, No. 155, December, 1948, p. 36 (TP244); Discussion, p. 37 (TP245).

The Importance of the Time Factor in the Study of the Textile Properties—Edward R. Schwarz. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 485.

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, 664. Discussion, 690.

Induction Period Calculator.

Induction Period Calculator—E. L. Korb. Published in ASTM BULLETIN, No. 153, August, 1948, p. 99 (TP191).

Industrial Water.

See **Water.**

Insulating Materials.

See **Electrical Insulating Materials; Refractories; Thermal Insulating Materials.**

Iron.

See also **Wrought Iron.**

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, 841. Disscusion, 853.

Corrosion of Iron and Steel. Report of Committee A-5, 104.

Fatigue and Static Load Tests of An Austenitic Cast Iron at Elevated Temperatures—W. Leighton Collins, 696. Discussion, 706.

Filler Metal.' Report of Joint Committee, 210.

Laboratory Corrosion Tests of Iron and Steel Pipes—G. A. Ellinger, L. J. Waldron, and S. B. Marzolf, 618.

Magnetic Properties. Report of Committee A-6, 125.

Iron Oxide Deposits.

Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Panel Discussion on Corrosion of Pressure Vessels—Richard C. Corey, 907. Discussion, 926.

Isotopes.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture—Paul C. Aebersold, 527.

K

Knock Characteristics.

Optional Use of Detonation Meter for Motor and Research Methods. Appendix VI, Report of Committee D-2, 328.

Knock Characteristics—*continued.*

Summary Report on Investigation of the Detonation Meter, Model 501. Appendix VIII, Report of Committee D-2, 340.

L

Lacquer.

See also **Electrical Insulating Materials.**
Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.

Laminated Plastics.

The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation Energy Theory—W. N. Findley, C. H. Adams, and W. J. Worley, 1217.
Low-Pressure Moldings and Laminates—T. Walter Noble. Published in ASTM BULLETIN, No. 151, March, 1948, p. 77 (TP85).

Lead.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.
Electrodeposited Metallic Coatings. Report of Committee B-8, 206.
Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, 815. Discussion, 825.
Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Lead Coatings.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.
Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Lead Scavenging.

Better Lead Scavenging Needed for Aviation Gasoline—E. A. Droegemueller. Published in ASTM BULLETIN, No. 154, October, 1948, p. 53 (TP211).

Lime.

Lime. Report of Committee C-7, 255.

Limestone.

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., 944. Discussion, 959.

Liquefied Petroleum Gasoline.

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases. Appendix V, Report of Committee D-2, 318.

Low-Temperature Test.

See also **Temperature, Effect of.**
Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.
Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.
Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives. Appendix IX, Report of Committee D-2, 354.
Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Lubricants.

Petroleum Products and Lubricants. Report of Committee D-2, 287. Supplementary Report, 298.
Petroleum Products as Related to Automotive Equipment, 1216.
Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease. Appendix I, Report of Committee D-2, 303.
Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.
The Thixotropic Characteristics of Lubricating Oil Greases—L. W. McLennan and G. H. Smith. Published in ASTM BULLETIN, No. 152, May, 1948, p. 71 (TP123).

A Worker-Consistometer for Lubricating Greases—S. A. McKee and H. S. White. Published in ASTM BULLETIN, No. 153, August, 1948, p. 90 (TP182); Discussion, p. 97 (TP189).

Lucite.

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

M

Magnesium.

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys—R. B. Smith. Published in ASTM BULLETIN, No. 151, March, 1948, p. 50 (TP58).

Die-Cast Metals and Alloys. Report of Committee B-6, 187.

The Effect of Speed of Testing on Magnesium-Base Alloys—Symposium on Speed of Testing—A. A. Moore, 1133.

Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 198.

Metals and Plastics—Robert G. Chollar. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP132); Discussion, p. 87 (TP139).

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion, 754.

Tension Testing of Magnesium Alloy Tubing, A. A. Moore, Appendix, Report of Committee B-7, 205.

What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Magnesium Alloy Tubing.

Tension Testing of Magnesium Alloy Tubing —A. A. Moore. Appendix, Report of Committee B-7, 205.

Magnesium Oxychloride Cements.

Magnesium Oxychloride Cements. Report of Committee C-2, 253.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements—Harry E. Chisholm. Published in ASTM BULLETIN, No. 154 October, 1948, p. 37 (TP195); Discussion, p. 39 (TP197).

Magnesium Sulfate Test.

Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests—Delmar L. Bloem. Published in ASTM BULLETIN, No. 152, May, 1948, p. 76 (TP128); Discussion, No. 153, August, 1948, p. 81 (TP173).

Magnetic Strain Gage.

A Stress-Strain Tester for Textiles Employing a Magnetic Strain Gage—J. W. Ballou. Published in ASTM BULLETIN, No. 150, January, 1948, p. 38 (TP10).

Magnetic Testing.

Magnetic Properties. Report of Committee A-6, 125.

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above The Endurance Limit—James B. Duke, 755. Discussion, 763.

Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Malleable Iron.

Malleable Iron Castings. Report of Committee A-7, 130.

Mason Jar Test

A Rapid Method of Testing Materials for the Alkali—Aggregate Reaction—Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete—D. O. Wolf and Theodore R. Smith, 1108. Discussion, 1115.

Masonry Materials.

Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, 1020. Discussion, 1040.

Mortars for Unit Masonry. Report of Committee C-12, 269.

Testing Surface Waterproofers—F. O. Anderegg. Published in ASTM BULLETIN, No. 156, January, 1949, p. 71 (TP33); Discussion, p. 76 (TP38).

Melting Point.

Summary of Softening Point and Melting Point Methods Used for Testing Rosins and Resins. Report of Subcommittee I, Committee D-17, 457.

Membership.

Annual Report of the Board of Directors, 35.

Metal Cleaners.

Soaps and Other Detergents. Report of Committee D-12, 431.

Metals in Lubricants.

Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives. Appendix IX, Report of Committee D-2, 354.

Metal Polish

Discussion of Paper on Measurement of Metal Polish Performance—F. E. Clark and R. C. Adams. Published in ASTM BULLETIN, No. 150, January, 1948, p. 49 (TP21).

Metal Powders.

Metal Powders and Metal Powder Products. Report of Committee B-9, 208.

Metallography.

See also **Copper, Brass, Steel,** etc.

Metallography. Report of Committee E-4, 510.

Summary of Proceedings of the Symposium on Metallography in Color, 942.

Methods of Testing.

(Papers and reports covering methods of analysis, sampling, testing, etc., are indexed under the materials and subjects covered by them.)

Methods of Testing. Report of Committee E-1, 498.

Mica.

Electrical Insulating Materials. Report of Committee D-9, 402.

Microhardness.

Metallography. Report of Committee E-4, 510.

Microorganisms.

Proposed Method for Identification of Types of Microorganisms in Industrial Waters. Appendix, Report of Committee D-19, 486.

Microscopic Test.

Proposed Method for Identification of Types of Microorganisms in Industrial Waters. Appendix, Report of Committee D-19, 486.

Mineral Aggregates.

Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Mineral Transformer Oil.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oils—Frank M. Clark and Edward L. Raab, 1201. Discussion, 1211.

Mode of Fracture.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—P. G. Jones and W. J. Worley, 648. Discussion 663.

Moisture Control.

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams—W. G. Holtz, 1240.

The Importance and Practical Use of Relative Density in Soil Mechanics—Donald M. Burmister, 1249.

Moisture Penetration.

Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, 1020. Discussion, 1040.

Mortar.

Determination of the Air Content of Mortars by the Pressure Method—Thomas G. Taylor. Published in ASTM BULLETIN, No. 155, December, 1948, p. 44 (TP252).

Lime. Report of Committee C-7, 255.

Mortars for Unit Masonry. Report of Committee C-12, 269.

Proposed Method of Test for Setting Time of Hydraulic Cement in Mortar. Appendix, Report of Committee C-1, 250.

N

Naval Stores.

Naval Stores. Report of Committee D-17, 451.

Summary of Softening Point and Melting

Point Methods Used for Testing Rosins and Resins, Report of Subcommittee I of Committee D-17, 457.

Nickel.

Abstracts of Papers on Cathode Nickel. Published in ASTM BULLETIN, No. 152, May, 1948:
- A Standard Diode for Radio Tube Cathode-Core-Material Approval Tests—Robert L. McCormack, p. 37.
- Testing Cathode Materials in Factory Production—J. T. Acker, p. 38.
- European Practice in the Manufacture of Cathodes—T. H. Briggs, p. 39.

Chemical Analysis of Metals. Report of Committee E-3, 505.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Nickel-Copper Alloys.

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Nomenclature.

See **Definitions.**

Non-Destructive Testing.

See also **Magnetic Testing; Radiographic Testing; Ultrasonic Testing.**

Radiographic Testing. Report of Committee E-7, 517.

Non-Ferrous Metals.

See **Alloys, non-Ferrous.**

Notch-Sensitivity.

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, 664. Discussion, 690.

Nuclear Physics.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture—Paul C. Aebersold, 527.

O

Oils.

See **Cutting Oils; Fuel Oils; Lubricants; Petroleum Products; Transformer Oils; Turbine Oils.**

Optical Materials.

Plastics as Optical Materials—H. R. Moulton. Published in ASTM BULLETIN, No. 151, March, 1948, p. 75 (TP83).

Organic Reagents.

Abstract of Papers on Organic Reagents for Metal Analysis. Published in ASTM Bulletin, No. 151, March, 1948:
- Organic Reagents for Gravimetric Analysis—John F. Flagg, p. 31.
- The Use of Organic Solvents in Metal Analysis—C. J. Rodden, p. 31.
- Organic Reagents in Colorimetric Analysis—John H. Yoe, p. 32.
- Organic Reagents for Volumetric Analysis—John F. Flagg, p. 32.
- Oxidation-Reduction Indicators—G. Frederick Smith, p. 33.

Oxidation Stability.

Induction Period Calculator—E. L. Korb. Publication in ASTM BULLETIN, No. 153, August, 1948, p. 99 (TP191).

Oxychloride Cement.

Magnesium Oxychloride Cements. Report of Committee C-2, 253.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements—Harry E. Chisholm. Published in ASTM BULLETIN, No. 154, October, 1948, p. 37 (TP195); Discussion, p. 39 (TP197).

P

Packing.

Shipping Containers. Report of Committee D-10, 415.

The Use of Test Track in Developing Good Packaging and Carloading Methods—A. N. Perry. Published in ASTM BULLETIN, No. 155, December, 1948, p. 38 (TP246).

Paint.

Apparatus and Factors in Salt Fog Testing, V. M. Darsey and W. R. Cavanagh. Appendix, Report of Committee B-3, 153.
The Gloss Measurement of Paint Finishes—Richard S. Hunter. Published in ASTM BULLETIN, No. 150, January, 1948, p. 72 (TP44).
Measurement of Transparency of Surface Coatings—J. Gross, R. H. Kienle, L. A. Melsheimer, and E. I. Stearns. Published in ASTM BULLETIN, No. 153, August, 1948, p. 73 (TP165).
Mechanical Operation of the Bird Film Applicator—A. E. Jacobsen and H. S. Jensen. Published in ASTM BULLETIN, No. 151, March, 1948, p. 95 (TP103).
A Method for the Routine Determination of Dry Hiding Power of White Paints—F. C. Schmutz and J. M. Gallagher. Published in ASTM BULLETIN, No. 153. August, 1948, p. 77 (TP169).
Paint—Servant of Industry (condensed version)—W. H. Lutz. Published in ASTM BULLETIN, No. 150, January, 1948, p. 14.
Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.
The Preparation of Uniform Films and Their Dependence on the Irregularities of the Base Surface—Stig Johanson. Published in ASTM BULLETIN, No. 155, December, 1948, p. 53 (TP261).
Report on the Evaluation of Surface Treatment of Steel Prior to Painting—Prepared by Arnold J. Eickhoff. Published in ASTM BULLETIN, No. 150, January, 1948, p. 77 (TP49).

Panels.

Method of Testing Building Constructions. Report of Committee E-6, 515.

Paper.

Electrical Insulating Materials. Report of Committee D-9, 402.
Load Concentration in Corrugated Paper—J. H. Toulouse and P. J. Barcus. Published in ASTM BULLETIN, No. 152, May, 1948, p. 77 (TP129).
Paper and Paper Products. Report of Committee D-6, 395.
Proposed Method of Wax Pick Test for Surface Strength of Paper. Appendix, Report of Committee D-6, 398.
Shipping Containers. Report of Committee D-10, 415.

Paperboard.

Paper and Paper Products. Report of Committee D-6, 395.

Papers and Publications.

Annual Report of the Board of Directors, 35.
Report of Administrative Committee on Papers and Publications. Appendix IV, Annual Report of the Board of Directors, 76.

Particle Size.

Methods of Testing. Report of Committee E-1, 498.

Paving Materials.

Road and Paving Materials. Report of Committee D-4, 383.
Research on Concrete Durability as Affected by Coarse Aggregate—Harold S. Sweet, 988. Discussion, 1017.

Petrographic Identification.

Petrographic Identification of Reactive Constituents in Concrete Aggregate—Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete—Bryant Mather, 1120. Discussion 1126.

Petroleum Products.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab, 1201. Discussion, 1211.
Summary of Proceedings of the Forum on Tractor Fuels, 1215.
Petroleum Products and Lubricants. Report of Committee D-2, 287. Supplementary Report, 298.
Photoelectric Measurement of a Color Index of Liquid Petroleum Products—Louis Lykken. Published in ASTM BULLETIN, No. 154, October, 1948, p. 68 (TP226).
Summary of Meeting on Petroleum Products as Related to Automotive Equipment, 1216. Optional Use of Detonation Meter for Motor

and Research Methods. Appendix VI, Report of Committee D-2, 328.

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases. Appendix V, Report of Committee D-2, 318.

Proposed Method of Analysis of Calcium and Barium Petroleum Sulfonates. Appendix XI, Report of Committee D-2, 367.

Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.

Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry. Appendix XII, Report of Committee D-2, 374.

Proposed Method of Test for Butadiene Dimer in Polymerization Grade Butadiene. Apendix III, Report of Committee D-2, 312.

Proposed Method of Test for Nonvolatile Residue of Polymerization Grade Butadiene. Appendix IV, Report of Committee D-2, 316.

Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease. Appendix I, Report of Committee D-2, 303.

Proposed Method of Test for Separation of Residue from Butadiene. Appendix II, Report of Committee D-2, 309.

Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives. Appendix IX, Report of Committee D-2, 354.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.

Summary Report on Investigation of the Detonation Meter, Model 501. Appendix VIII, Report of Committee D-2, 340.

Pharmaceutical Containers.

Glass and Glass Products. Report of Committee C-14, 270.

pH Value.

Methods of Testing. Report of Committee E-1, 498.

Photometric Methods.

Chemical Analysis of Metals. Report of Committee E-3, 505.

Photoelectric Measurement of a Color Index of Liquid Petroleum Products—Louis Lykken. Published in ASTM BULLETIN, No. 154, October, 1948, p. 68 (TP226).

Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry. Appendix XII, Report of Committee D-2, 374.

Summary of Proceeding of the Symposium on Metallography in Color, 942.

Pigments.

Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.

Pine Oil.

Naval Stores. Report of Committee D-17, 451.

Pipe and Pipe Materials.

Clay Pipe. Report of Committee C-4, 254.

Laboratory Corrosion Tests of Iron and Steel Pipes—G. A. Ellinger, L. J. Waldron and S. B. Marzolf, 618.

Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Placement Conditions.

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams—W. G. Holtz, 1240.

Pitch.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.

Plastics.

See also **Electrical Insulating Materials.**

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Discussion of Paper on the Ignition Temperature of Rigid Plastics. Published in ASTM BULLETIN, No. 151, March, 1948, p. 66 (TP74).

Plastics—*continued.*

Discussion of Paper on the Resistance to Impact Loading of Plastics. Published in ASTM BULLETIN, No. 153, August, 1948, p. 59 (TP151); No. 154, October, 1948, p. 45 (TP203).

The Effect of Fungi and Humidity on Plastics—John Leutritz, Jr. Published in ASTM BULLETIN, No. 152, May, 1948, p. 88 (TP140).

Effect of Speed of Test Upon Strength Properties of Plastics—Symposium on Speed of Testing—A. G. H. Dietz, W. J. Gailus, and S. Yurenka, 1160. Discussion, 1187.

The Effect of Temperature on the Creep of two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation Energy Theory—W. N. Findley, C. H. Adams, and W. J. Worley, 1217.

Electrical Insulating Materials. Report of Committee D-9, 402.

Electrical Properties of Plastics—A. J. Warner. Published in ASTM BULLETIN, No. 153, August, 1948, p. 60 (TP152).

An Improved Low-Temperature Brittleness Test—E. F. Smith and G. J. Dienes. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204); Discussion, p. 49 (TP207).

An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster, 628. Discussion, 645.

Low-Pressure Molding and Laminates—T. Walter Noble. Published in ASTM BULLETIN, No. 151, March, 1948, p. 77 (TP85).

The Mechanism of Plasticization in Plastics—J. Kenneth Craver. Published in ASTM BULLETIN, No. 152, May, 1948, p. 90 (TP142); Discussion, p. 92 (TP144).

Metals and Plastics—Robert G. Chollar. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP132); Discussion, p. 87 (TP139).

A New Interpretation of Heat Distortion Data of Plastics—L. S. Moody and K. B. Goldblum. Published in ASTM BULLETIN, No. 150, January, 1948, p. 55 (TP27).

Plastics as Optical Materials—H. R. Moulton. Published in ASTM BULLETIN, No. 151, March, 1948, p. 75 (TP83).

Plastics. Report of Committee D-20, 490.

Recommendations Affecting Standards on Plastics. Appendix, Report of Committee D-20, 494.

Some Factors Affecting Adhesive Bond Formation—H. P. Meissner and E. W. Merrill. Published in ASTM BULLETIN, No. 151, March, 1948, p. 80 (TP88).

Tensile Stress-Strain Relationships of Laminated Plastics for Small Strains—J. J. Lamb and B. M. Axilrod. Published in ASTM BULLETIN, No. 151, March, 1948, p. 59 (TP67).

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C.—F. E. Piech and W. E. Gloor. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers—Charles J. Seiler and A. D. McLaren. Published in ASTM BULLETIN, No. 155, December, 1948, p. 50 (TP258).

Plexiglas.

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Plywood.

Wood. Report of Committee D-7, 400.

Polarographic Methods.

Chemical Analysis of Metals. Report of Committee E-3, 505.

Poles.

Wood. Report of Committee D-7, 400.

Polish

Discussion of Paper on Measurement of Metal Polish Performance—F. E. Clark and R. C. Adams. Published in ASTM BULLETIN, No. 150, January, 1948, p. 49 (TP21).

Polymers.

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers—Charles J. Seiler and A. D. McLaren.

Published in ASTM BULLETIN, No. 155, December, 1948, p. 50 (TP258).

Porcelain Enamel.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Prebatched Mixtures.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete—W. H. Price and J. W. Robison, 962. Discussion, 968.

President's Address and Talks.

If Doctor Marburg Came Back Today, Annual Address by the President, T. A. Boyd, 26.

Research—Everybody's Doing It Now—T. A. Boyd. Published in ASTM BULLETIN, No. 152, May, 1948, p. 53 (TP105).

Pressure Method.

Determination of the Air Content of Mortars by the Pressure Method—Thomas G. Taylor. Published in ASTM BULLETIN, No. 155, December, 1948, p. 44 (TP252).

Pressure Vessels.

Panel Discussion on Corrosion of Pressure Vessels:

Station Design and Material Composition as Factors in Boiler Corrosion—R. B. Donworth, 897. Discussion, 905.

Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, 907. Discussion, 926.

Protective Coatings.

See also **Paint.**

An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster, 628. Discussion, 645.

Measurement of Transparency of Surface Coatings—J. Gross, R. H. Kienle, L. A. Melsheimer, and E. I. Stearns. Published in ASTM BULLETIN, No. 153, August, 1948, p. 73 (TP165).

Proving Ring.

Dynamic Calibration Method Uses Modified Proving Ring—R. C. A. Thurston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 50 (TP208).

Q

Quality Control.

Quality Control of Materials. Report of Committee E-11, 523.

Symposium on Usefulness and Limitations of Samples:

Introduction—H. F. Dodge, 857.

Sampling and Its Uncertainties—S. S. Wilks, 859. Discussion, 876.

On Variation in Materials, Testing, and Sample Sizes—Leslie E. Simon, 877. Discussion, 882.

The Amount of Inspection as a Function of Control of Quality—G. R. Gause, 886. Discussion, 894.

Quartz Tube Combustion.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Quicklime.

Lime. Report of Committee C-7, 255.

R

Radioactive Material.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture—Paul C. Aebersold, 527.

Radiography.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts—W. W. Buechner, R. J. Van de Graaff, H. Feshbach, E. A. Burrill, A. Sperduto, and L. R. McIntosh. Published in ASTM BULLETIN, No. 155, December, 1948, p. 54 (TP262).

Frankford Arsenal Experience with High-Speed Radiography—E. R. Thilo. Published in ASTM BULLETIN, No. 150, January, 1948, p. 69 (TP41).

Radiography—*continued.*

Metallography. Report of Committee E-4, 510.

One-Millionth Second Radiography and Its Development—Charles M. Slack and Louis F. Ehrke. Published in ASTM BULLETIN, No. 150, January, 1948, p. 59 (TP31).

Radiographic Testing. Report of Committee E-7, 517.

Radio Tube Materials.

Electrical-Heating Resistance, and Related Alloys. Report of Committee B-4, 176.

Abstracts of Paper on Cathode Nickel. Published in ASTM BULLETIN, No. 152, May, 1948:

A Standard Diode for Radio Tube Cathode-Core-Material Approval Tests—Robert L. McCormack, p. 37.

Testing Cathode Materials in Factory Production—J. T. Acker, p. 38.

European Practice in the Manufacture of Cathodes—T. H. Briggs, p. 39.

Rate of Loading.

See **Speed of Testing.**

Rayon.

Some Applications of Stress-Strain Measurements to the Determination of Fiber Properties—Arnold M. Sookne. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 498.

Textile Materials. Report of Committee D-13, 434.

Reactive Materials in Concrete.

Symposium on Methods and Procedures Used in Identifying Reactive Materials in Concrete:

Introduction, 1055.

Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Thomas E. Stanton, 1057. Discussion, 1063.

Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Bailey Tremper, 1067.

Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Richard C. Mielenz and Leslie P. Witte, 1071. Discussion, 1104.

A Rapid Method of Testing Materials for the Alkali-Aggregate Reaction—D. O. Woolf and Theodore R. Smith, 1108. Discussion, 1115.

Petrographic Identification of Reactive Constituents in Concrete Aggregate—Bryant Mather, 1120. Discussion, 1126.

Reactivity.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method—J. Jonakin, P. Cohen, R. Corey, and B. Jain, 1269. Discussion, 1290.

Refractories.

Refractories. Report of Committee C-8, 256.

Relative Density.

The Importance and Practical Use of Relative Density in Soil Mechanics—Donald M. Burmister, 1249.

Research.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.

Report of Administrative Committee on Research. Appendix III, Annual Report of the Board of Directors, 73.

Research—Everybody's Doing It Now—T. A. Boyd. Published in ASTM BULLETIN, No. 152, May, 1948, p. 53 (TP105).

Resin.

Summary of Softening Point and Melting Point Method Used for Testing Rosins and Resins, Report of Subcommittee I, Committee D-17, 457.

Road Materials.

See also **Aggregates; Brick; Cement; Concrete; Soils.**

Road and Paving Materials. Report of Committee D-4, 383.

Soils for Engineering Purposes. Report of Committee D-18, 476.

Roofing Materials.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.

Fire Tests of Materials and Construction. Report of Committee E-5, 512.

Rosin.

Naval Stores. Report of Committee D-17, 451.

Rossi-Peakes Flow Tester.

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C.—F. E. Piech and W. E. Gloor. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Rubber Products.

Nontoxic Rubber for Gas Masks—S. H. Katz, 1293. Discussion, 1299.

Proposed Method of Measuring Low-Temperature Stiffening of Rubber ond Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.

Rubber and Rubber-Like Materials. Report of Committee D-11, 417.

Rubber-Like Materials.

Measurement of Volume Resistivity of Electrically Conducting Rubber-Like Material—R. F. Miller. Published in ASTM BULLETIN, No. 151, March, 1948, p. 91 (TP99).

S

Salt Spray Tests.

See also **Corrosion.**

Apparatus and Factors in Salt Fog Testing, V. M. Darsey and W. R. Cavanagh. Appendix, Report of Committee B-3, 153.

Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.

Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium. Appendix, Report of Committee B-6, 191.

Samples, Usefulness and Limitations of.

Quality Control of Materials. Report of Committee E-11, 523.

Symposium on Usefulness and Limitations of Samples:

Introduction—H. F. Dodge, 857.

Sampling and Its Uncertainties—S. S. Wilks, 859. Discussion, 876.

On Variation in Materials, Testing, and Sample Sizes—Leslie E. Simon, 877. Discussion, 882.

The Amount of Inspection as a Function of Control of Quality—G. R. Gause, 886. Discussion, 894.

Sampling of Steam.

Proposed Method for the Sampling of Steam. Published in ASTM BULLETIN, No. 151, March, 1948, p. 34.

Sand.

Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests—Delmar L. Bloem. Published in ASTM BULLETIN, No. 152, May, 1948, p. 76 (TP128). Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 81 (TP173).

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., 944. Discussion 959.

Sanford E. Thompson Award.

Summary of Proceedings of the Fifty-first Annual Meeting, 22. Report of Committee C-9, 263.

Service Tests.

Service Wearing Tests—E. N. Ditton. Published in ASTM BULLETIN, No. 152, May, 1948, p. 93 (TP145).

Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, May, 1948:

An Evaluation of Inhibited Turbine Oils Versus Uninhibited Turbine Oils for Turbine Lubrication—M. D. Baker, p. 59 (TP111).

Service Experience with Turbine Oils—C. L. Pope and O. V. Sprague, p. 62 (TP114).

The Care of the Lubricant and Maintenance of the Lubricating System for Central Station Turbine Equipment—Ernest F. Walsh, p. 64 (TP116).

Steam Turbines and Their Lubrication—C. D. Wilson, p. 66 (TP118).

Setting Time.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cement—Harry E. Chisholm. Published in ASTM BULLETIN, No. 154, October, 1948, p. 37 (TP195); Discussion, p. 39 (TP197).

Proposed Method of Test for Setting Time of Hydraulic Cement in Mortar. Appendix, Report of Committee C-1, 250.

Sheathing.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.

Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, 815. Discussion, 825.

Shellac.

Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.

Sherardized Coatings.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Shingles.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.

Shipping Containers.

Load Concentration in Corrugated Paper—J. H. Toulouse and P. J. Barcus. Published in ASTM BULLETIN, No. 152, May, 1948, p. 77 (TP129).

Shipping Containers. Report of Committee D-10, 415.

Ship Plates.

Summary of the Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Significance of Tests.

Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Silver.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.

Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, 815. Discussion, 825.

Simulated Service Testing.

Engine Antifreezes. Report of Committee D-15, 448.

Methods of Testing Building Constructions. Report of Committee E-6, 515.

Report of Administrative Committee on Simulated Service Testing. Appendix VI, Annual Report of the Board of Directors, 86.

The Use of Test Track in Developing Good Packaging and Carloading Methods—A. N. Perry. Published in ASTM BULLETIN, No. 155, December, 1948, p. 38 (TP246).

Slate.

Natural Building Stones. Report of Committee C-18, 275.

Soaps and Detergents.

Soaps and Other Detergents. Report of Committee D-12, 431.

Sodium and Potassium in Lubricants.

Proposed Method of Test for Sodium in Lubricating Oils and Lubricating Oil Additives. Appendix IX, Committee D-2, 354.

Softening Point.

Summary of Softening Point and Melting Point Methods Used for Testing Rosins and Resins, Report of Subcommittee I, of Committee D-17, 457.

Soils.

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams—W. C. Holtz, 1240.

The Importance and Practical Use of Relative Density in Soil Mechanics—Donald M. Burmister, 1249.

Soils for Engineering Purposes. Report of Committee D-18, 476.

Solder.

Non-Ferrous Metals and Alloys. Report of Committee B-2, 143.

Soundness Tests.

Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests—Delmar L. Bloem. Published in ASTM BULLETIN, No. 152, May, 1948, p. 76 (TP128). Discussion published in ASTM BULLETIN, No. 153, August, 1948, p. 81 (TP173).

Specific Gravity.

A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range—Richard C. Weatherwax, E. C. O. Erickson, and Alfred J. Stamm. Published in ASTM BULLETIN, No. 153, August, 1948, p. 84 (TP176).

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases. Appendix V, Report of Committee D-2, 318.

Spectrographic Analysis.

The Spectrochemical Analysis of Cements and Other Mineral Products—M. F. Hasler, C. E. Harvey, and F. W. Barley, Jr., 944. Discussion, 959.

Spectrographic Analysis. Report of Committee E-2, 503.

Speed of Testing.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—P. G. Jones and W. J. Worley, 648. Discussion, 663.

Methods of Testing. Report of Committee E-1, 498.

Symposium on Speed of Testing:

  Introduction, 1129.

  Testing Speed Limitations for Committee A-1, Specifications for Steel—Lawford H. Fry, 1130.

  The Effect of Speed of Testing on Magnesium-Base Alloys—A. A. Moore, 1133.

  Speed of Testing of Wood: Factors in Its Control and Its Effect on Strength—L. J. Markwart and J. A. Liska, 1139.

  Effect of Speed of Test Upon Strength Properties of Plastics—A. G. H. Dietz, W. J. Gailus, and S. Yurenka, 1160. Discussion, 1187.

  Methods and Equipment for Controlling Speed of Testing—Lawrence K. Hyde, 1191. Discussion, 1200.

Stability of Steels.

See also **Steel.**

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.

Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Standardization.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Summary of Proceedings of the Fifty-first Annual Meeting, 1.

Statistical Analysis.

Quality Control of Materials. Report of Committee E-11, 523.

Symposium on Usefulness and Limitations of Samples.

  Introduction—H. F. Dodge, 857.

  Sampling and Its Uncertainties—S. S. Wilks, 859. Discussion, 876.

  On Variation in Materials, Testing, and Sample Sizes—Lesslie E. Simon, 877. Discussion, 882.

  The Amount of Inspection as a Function of Control of Quality—G. R. Gause, 886. Discussion, 894.

Steel.

Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, 610.

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending—Harry Majors, Jr. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).

Steel—*continued.*

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, 841. Discussion, 853.

Chemical Analysis of Metals. Report of Committee E-3, 505.

Corrosion of Iron and Steel. Report of Committee A-5, 104.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, **212.**

Electrodeposited Metallic Coatings, Report of Committee B-8, 206.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals—P. G. Jones and W. J. Worley, 648. Discussion 663.

Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, 591. Discussion, 608.

Field Tests and Inspections of Bar and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Filler Metal. Report of Joint Committee, 210.

High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, 555. Discussion, 589.

An Hypothesis for the Determination of Cumulative Damage in Fatigue—F. E. Richart, Jr., and N. M. Newmark, 767. Discussion 799.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.

An Investigation of the Possibilities or Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel—Robert C. McMaster, 628. Discussion 645.

Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys. Report of Committee A-10, 134.

Laboratory Corrosion Tests of Iron and Steel Pipes—G. A. Ellinger, L. J. Waldron, and S. B. Marzolf, 618.

Magnetic Properties. Report of Committee A-6, 125.

Metallography. Report of Committee E-4, 510.

Metals and Plastics—Robert G. Chollar. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP132); Discussion, p. 87 (TP139).

Recommendations Affecting Standards on Steel. Appendix, Report of Committee A-1, 95.

Report on the Evaluation of Surface Treatment of Steel Prior to Painting—Prepared by Arnold J. Eickhoff. Published in ASTM BULLETIN, No. 150, January, 1948, p. 77 (TP49).

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel—T. J. Dolan and C. S. Yen, 664. Discussion, 690.

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above The Endurance Limit—James B. Duke, 755. Discussion, 763.

Steel. Report of Committee A-1, 90.

Summary of the Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Testing Speed Limitations for Committee A-1 Specifications for Steel—Symposium on Speed of Testing—Lawford H. Fry, 1130.

A Visual Rating System for Rusted Steel Specimens—Harry L. Faigen. Published in ASTM BULLETIN, No. 154, October, 1948, p. 39 (TP197).

Stiffness.

Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.

Stone.

See also **Aggregates.**

Natural Building Stones. Report of Committee C-18, 275.

Stress Analysis.

Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Stress-Strain Tester.

A Stress-Strain Tester for Textiles Employing a Magnetic Strain Gage—J. W. Ballou. Published in ASTM BULLETIN, No. 150. January, 1948, p. 38 (TP10).

Structural Units.

Methods of Testing Building Constructions. Report of Committee E-6, 515.

Sulfonated Oils.

Proposed Method of Analysis of Calcium and Barium Petroleum Sulfonates. Appendix XI, Report of Committee D-2, 367.

Sulfur.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab, 1201. Discussion, 1211.

Proposed Method of Test for Sulfur in Petroleum Oils by Quartz Tube Combustion. Appendix VII, Report of Committee D-2, 332.

Sulfur Cements.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Summary of Proceedings.

Summary of Proceedings of the Fifty-first Annual Meeting, 1.

Surface Strength of Paper.

Proposed Method of Wax Pick Test for Surface Strength of Paper. Appendix, Report of Committee D-6, 398.

Surface Waterproofers.

Testing Surface Waterproofers—F. O. Anderegg. Published in ASTM BULLETIN, No. 156, January, 1949, p. 71 (TP33); Discussion, p. 76 (TP38).

Symbols.

Symbols and Nomenclature for Fatigue Testing. Published in ASTM BULLETIN, No. 153, August, 1948, p. 36.

Synthetic Detergents.

Soaps and Other Detergents. Report of Committee D-12, 431.

Synthetic Fibers.

Application of Synthetic Fibers, Including Glass, in the Textile Industry (Condensed version)—Rene Bouvet. Published in ASTM BULLETIN, No. 150, January, 1948, p. 20.

Synthetic Rubber.

Rubber and Rubber-Like Materials. Report of Committee D-11, 417.

T

Tall Oil.

Naval Stores. Report of Committee D-17, 451.

Tar.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.

Road and Paving Materials. Report of Committee D-4, 383.

Temperature, Effect of.

The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation Energy Theory—W. N. Findley, C. H. Adams, and W. J. Worley, 1217.

Effect of Temperature on the Properties of Metals. Report of Joint Committee, 212.

Electrical-Heating, Resistance, and Related Alloys. Report of Committee B-4, 176.

Engine Antifreezes. Report of Committee D-15, 448.

Fatigue and Static Load Tests of An Austenitic Cast Iron at Elevated Temperatures—W. Leighton Colling, 696. Discussion, 706.

High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing—J. W. Freeman, Howard C. Cross, E. E. Reynolds, and Ward F. Simmons, 555. Discussion, 589.

Temperature, Effect of—*continued.*

An Improved Low-Temperature Brittleness Test—E. F. Smith and S. J. Diener. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46.

Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.

Stability of Steels as Affected by Temperature (Project No. 29), J. J. Kanter and E. A. Sticha, Appendix II, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion 754.

Temperature Measurements.

Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.

Templin Award.

Richard L. Templin Award. Summary of Proceedings of the Fifty-first Annual Meeting, 16.

Tension Testing.

Effect of Fatigue on Tension-Impact Resistance—William H. Hoppmann II. Published in ASTM BULLETIN, No. 155, December, 1948, p. 36 (TP244); Discussion, p. 37 (TP245).

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead—G. R. Gohn and W. C. Ellis, 801. Discussion, 825.

The Effect of Speed of Testing on Magnesium Base Alloys—Symposium on Speed of Testing—A. A. Moore, 1133.

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form—H. L. Burghoff and A. I. Blank, 709. Discussion, 733.

High Temperature Bolting Materials (Project No. 16), Ernest L. Robinson. Appendix I, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 214.

An Improved Low-Temperature Brittleness Tester—E. F. Smith and G. J. Dienes. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204); Discussion p. 49 (TP207).

Influence of Small Percentages of Silver on The Tensile Strength of Extruded Lead Sheathing—Howard S. Phelps, Frank Kahn, and William P. Magee, 815. Discussion, 825.

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys—John C. McDonald, 737. Discussion, 754.

Tensile Stress-Strain Relationships of Laminated Plastics for Small Strains—J. J. Lamb and B. M. Axilrod. Published in ASTM BULLETIN, No. 151, March, 1948, p. 59 (TP67).

Tension Testing of Magnesium Alloy, A. A. Moore. Appendix, Report of Committee B-7, 205.

Testing Speed Limitations for Committee A-1 Specifications for Steel—Symposium on Speed of Testing—Lawford H. Fry, 1130.

Test Track.

The Use of Test Track in Developing Good Packaging and Carloading Methods—A. N. Perry. Published in ASTM BULLETIN, No. 155, December, 1948, p. 38 (TP246).

Testing Apparatus.

Methods of Testing. Report of Committee E-1, 498.

Testing, Physical Methods of.

(Papers and reports covering methods of analysis, sampling, testing, etc., are indexed under the materials and subjects covered by them.)

Testing, Speed of

See **Speed of Testing.**

Tetraethyl Lead.

Better Lead Scavenging Needed for Aviation Gasoline—E. A. Droegemueller. Published in ASTM BULLETIN, No. 154, October, 1948, p. 53 (TP211).

Textile Materials.

Application of Synthetic Fibers, Including Glass, in the Textile Industry (Condensed version)—Rene Bouvet. Published in ASTM BULLETIN, No. 150, January, 1948, p. 20.

The Importance of the Time Factor in the Study of Textile Properties—Edward R. Schwarz. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 485.

Measurement and Elimination of Inertia Effects in Textile Testing Equipment by Means of Electric Strain Gages—Herbert F. Schiefer. Published in ASTM BULLETIN, No. 150, January, 1948, p. 43 (TP15).

Recommendations Affecting Standards on Textile Materials. Appendix, Report of Committee D-13, 440.

Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter—Seaman J. Tanenhaus and Gerald Winsont. Published in ASTM BULLETIN, No. 154, October, 1948, p. 74 (TP232).

Service Wearing Tests—E. N. Ditton. Published in ASTM BULLETIN, No. 152, May, 1948, p. 93 (TP145).

Some Applications of Stress-Strain Measurements to the Determination of Fiber Properties—Arnold M. Sookne. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 498.

The Stress-Strain Diagram as a Tool in Textile Research—Walter J. Hamburger. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 491.

A Stress-Strain Tester for Textiles Employing a Magnetic Strain Gage—J. W. Ballou. Published in ASTM BULLETIN, No. 150, January, 1948, p. 38 (TP10).

Symposium on Flammability. Published in ASTM BULLETIN, No. 150, January, 1948:

Flammability of Textiles for Garment Purposes—Frederic Bonnet, p. 29 (TP1).

Flameproofing in Theory and Practice——R. W. Little, p. 34 (TP6).

The Retailer's Viewpoint on the Flammability of Textiles—Charles W. Dorn, p. 36 (TP8).

Textile Materials. Report of Committee D-13, 434.

Thermal Insulating Materials.

Thermal Insulating Materials. Report of Committee C-16, 271.

A Versatile Calorimeter for Specific Heat Determinations—Harry Hill and Raymond M. Bell. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Thermometers.

Methods of Testing. Report of Committee E-1, 498.

Proposed Method of Measuring the Temperature of Petroleum and Petroleum Products. Appendix X, Report of Committee D-2, 357.

Thermostat Metals.

Electrical-Heating, Resistance, and Related Alloys. Report of Committee B-4, 176.

Thixotrapy.

The Thixotropic Characteristics of Lubricating Oil Greases—L. W. McLennan and G. H. Smith. Published in ASTM BULLETIN, No. 152, May, 1948, p. 71 (TP123).

Timber.

See **Wood.**

Tin.

A Review of the Tin Situation, Marshall W. Tuthill. Appendix, Report of Committee B-2, 146.

What's in an Alloy?—John C. Kiszka. Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Toluene.

Proposed Method of Test for Benzene and Toluene by Ultraviolet Spectrophotometry. Appendix XII, Report of Committee D-2. 374.

Torsion Test.

Proposed Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus. Appendix, Report of Committee D-11, 425.

Toxid Accelerators.

Nontoxic Rubber for Gas Masks—S. H. Katz, 1293. Discussion, 1299.

Tractor Fuels.

Summary of Proceedings of the Forum on Tractor Fuels, 1215.

Traffic Paint.
See **Paint.**

Transformer Oil.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil—Frank M. Clark and Edward L. Raab, 1201. Discussion, 1211.

Electrical Insulating Materials. Report of Committee D-9, 402.

Transparency.

Measurement of Transparency of Surface Coatings—J. Gross, R. H. Kienle, L. A. Melsheimer, and E. I. Stearns. Published in ASTM BULLETIN, No. 153, August, 1948, p. 73 (TP165).

Tubes.

Field Tests and Inspections of Bar and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Turbine Oils.

Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, May, 1948.

An Evaluation of Inhibited Turbine Oils Versus Uninhibited Turbine Oils for Turbine Lubrication—M. D. Baker, p. 59 (TP111).

Service Experience with Turbine Oils—C. L. Pope and O. V. Sprague, p. 62 (TP114).

The Care of the Lubricant and Maintenance of the Lubricating System for Central Station Turbine Equipment—Ernest F. Walsh, p. 64 (TP116).

Steam Turbines and Their Lubrication—C. D. Wilson, p. 66 (TP118).

U

Ultimate Consumer Goods.

Report of Administrative Committee on Ultimate Consumer Goods. Appendix V, Annual Report of the Board of Directors, 84.

Ultrasonic Testing.

Radiographic Testing. Report of Committee E-7, 517.

Uranium.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture—Paul C. Aebersold, 527.

V

Varnish.

Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.

Vegetable Fiber Gaskets

Fungus Resistance of Treated Vegetable Fiber Gaskets—Sigmund Berk. Published in ASTM BULLETIN, No. 150, January, 1948, p. 46 (TP18).

Viscosity.

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C.—F. E. Piech and W. E. Gloor. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Volume Correction Tables.

Proposed Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases. Appendix V, Report of Committee D-2, 318.

Volume Determinations.

An Instrument for Determining the Volume and Bulk Density of Granular Materials—William E. Gross and John C. Goshorn. Published in ASTM BULLETIN, No. 151, March, 1948, p. 55 (TP63).

Measurement of Volume Resistivity of Electrically Conducting Rubber-Like Material—R. F. Miller. Published in ASTM BULLETIN, No. 151, March, 1948, p. 91 (TP99).

W

Walls.

Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, 1020. Discussion, 1040.

Water.

Industrial Water. Report of Committee D-19, 480.
Panel Discussion on Corrosion of Pressure Vessels:
  Station Design and Material Composition as Factors in Boiler Corrosion—R. B. Donworth, 897. Discussion, 905.
  Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Richard C. Corey, 907. Discussion, 926.
Proposed Method for Identification of Types of Microorganisms in Industrial Waters. Appendix, Report of Committee D-19, 486.
Proposed Method for the Sampling of Steam. Published in ASTM BULLETIN, No. 151, March, 1948, p. 34.

Waterproofing Materials.

Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 401.
Factors in the Resistance of Brick Masonry Walls to Moisture Penetration—C. C. Connor, 1020. Discussion, 1040.
Testing Surface Waterproofers—F. O. Anderegg. Published in ASTM BULLETIN, No. 155, January, 1949, p. 71 (TP33); Discussion, p. 76 (TP38).

Wax Pick Test.

Proposed Method of Wax Pick Test for Surface Strength of Paper. Appendix, Report of Committee D-6, 398.

Wear Tests.

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces—J. N. Good and Douglas Godfrey, 841. Discussion, 853.
Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter—Seaman J. Tanenhaus and Gerald Winston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 74 (TP232).
Service Wearing Tests—E. N. Ditton. Published in ASTM BULLETIN, No. 152, May, 1948, p. 93 (TP145).

Weathering.

See also **Corrosion.**
Atmospheric Durability of Steel Containing Nickel and Copper—Additional Exposure Data—N. B. Pilling and W. A. Wesley, 610.
Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 151.
Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels—H. R. Copson, 591. Discussion, 608.
Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.
Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.
Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.
Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 279.
Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 per cent Silicon and 0.5 per cent Magnesium or Containing 8 per cent Magnesium Appendix, Report of Committee B-6, 191.

Weighted Needles.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements—Harry E. Chisholm. Published in ASTM BULLETIN, No. 154, October, 1948, p. 37 (TP195); Discussion, p. 39 (TP197).

Welds.

Filler Metal. Report of Joint Committee, 210.
Radiographic Testing. Report of Committee E-7, 517.

White Wares.

Report of Administrative Committee on Standards. Appendix II, Annual Report of the Board of Directors, 69.

Windshields.

Automotive Glazing with Plastics—George B. Watkins and Joseph D. Ryan. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Wire.

See also **Steel.**

Electrical-Heating, Resistance, and Related Alloys. Report of Committee B-4, 176.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Wires for Electrical Conductors. Report of Committee B-1, 138.

Wood.

Fire Tests of Materials and Construction. Report of Committee E-5, 512.

A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range—Richard C. Weatherwax, E. C. O. Erickson and Alfred J. Stamm. Published in ASTM BULLETIN, No. 153, August, 1948, p. 84 (TP176).

Speed of Testing of Wood: Factors in its Control and Its Effect on Strength—Symposium on Speed of Testing—L. J. Markwardt and J. A. Liska, 1139.

Wood. Report of Committee D-7, 400.

Wool.

Some Application of Stress-Strain Measurements to the Determination of Fiber Properties—Arnold M. Sookne. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 498.

Textile Materials. Report of Committee D-13, 434.

Wrought Iron.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5, 108.

Magnetic Properties. Report of Committee A-6, 125.

Wyzenbeek Test Meter.

Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter—Seaman J. Tanenhaus and Gerald Winston. Published in ASTM BULLETIN, No. 154, October, 1948, p. 74 (TP232).

X

X-ray.

See **Radiography.**

Z

Zinc.

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending—Harry Majors, Jr. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).

Die-Cast Metals and Alloys. Report of Committee B-6, 187.

Metals and Plastics—Robert G. Chollar. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP 132); Discussion, p. 87 (TP139).

What's in An Alloy?—John C. Kiska. Published in ASTM BULLETIN, No. 151, March, 1948, p. 50 (TP58).

Zinc Coatings.

The Action of "Chromic Acid" on Zinc Coatings—D. J. Swaine. Published in ASTM BULLETIN, No. 154, October, 1948, p. 52 (TP210). Discussion, published in ASTM BULLETIN, No. 165, January, 1949, p. 49 (TP11).

Corrosion of Iron and Steel. Report of Committee A-5, 104.

Electrodeposited Metallic Coatings. Report of Committee B-8, 206.

Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars, and Their Products. Report of Subcommittee XVI, Committee A-5, 119.

Field Tests of Wire and Wire Products. Report of Wire Inspection Committee, Committee A-5, 114.

Inspection of Black and Galvanized Sheets. Report of Subcommittee XIV, Committee A-5. 108.

AUTHOR INDEX

VOLUME 48

A

Aber, W. C.

A Method of Calibrating Extensometers. Published in ASTM BULLETIN, No. 155, December, 1948, p. 33 (TP241).

Acker, J. T.

Testing Cathode Materials in Factory Production (condensed version). Published in ASTM BULLETIN, No. 152, May, 1948, p. 38.

Adams, C. H.

The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation Energy Theory, 1217.
Discussion, 1188.

Adams, R. C.

Discussion 905.

Aebersold, Paul C.

Isotopes and Their Application in the Field of Industrial Materials. Twenty-second Edgar Marburg Lecture, 527.

Allen, C. W.

Influence of Mineral Aggregates on the Strength and Durability of Concrete—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Allen, J. J.

Discussion, 1299.

Alquist, F. N.

Discussion, 927.

Anderegg, F. O.

Testing Surface Waterproofers. Published in ASTM Bulletin, No. 156, January, 1949, p. 71 (TP129); Discussion, p. 76 (TP38).
Discussion, 969, 1049.

Anderson, Marshall

Grease—An Oil Storehouse for Bearings—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Greases, 1214.

Anderson, Arthur B.

The Complexity of Tractor Fuels and Taxation—Summary of Proceedings of Forum on Tractor Fuels, 1215.

Ashworth, J. A.

Core Loss Test for Narrow Silicon Steel Strip—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Axilrod, B. M.

Tensil Stress-Strain Relationships of Laminated Plastics for Small Strains. Published in ASTM BULLETIN, No. 151, March, 1948, p. 59 (TP67).

B

Baker, M. D.

An Evaluation of Inhibited Turbine Oils Versus Uninhibited Turbine Oils for Turbine Lubrication—Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, May, 1948, p. 59 (TP111).
Discussion, 933.

Ballou, J. W.

A Stress-Strain Tester for Textiles Employing a Magnetic Strain Gage. Published in ASTM BULLETIN, No. 150, January, 1948, p. 38 (TP10).

Barcus, P. J.

Load Concentration in Corrugated Paper. Published in ASTM BULLETIN, No. 152, May, 1948, p. 77 (TP129).

Barger, E. L.

The Farmers' Interest in Tractor Fuels—Summary of Proceedings of Forum on Tractor Fuels, 1215.

Barley, F. W., Jr.

The Spectrochemical Analysis of Cements and Other Mineral Products, 944.

Barton, J. F.

Recent Development in Portland Cement (With a Thumbnail Sketch of the Industry) (condensed version) Published in ASTM BULLETIN, No. 150, January, 1948, p. 18.

Barnett, R. S.

Laboratory Performance Tests for Antifriction Bearing Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Grease, 1214.

Bech, P. A.

Discussion, 855.

Beckwith, O. P.

Discussion, 883, 894.

Beiler, A. C.

Variation of Core Loss and Permeability of Electrical Grade Silicon Sheet Steel—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Bell, Raymond M.

A Versatile Calorimeter for Specific Heat Determinations. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Belt, John R.

Factors Affecting Simulated Service Tests of Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Greases, 1214.

Benford, J. R.

Microscope Optics for Color Metallography—Summary of Proceedings of the Symposium on Metallography in Color, 942.

Bennett, J. A.

Discussion, published in ASTM BULLETIN, No. 155, December, 1948, p. 37 (TP245). Discussion, 734.

Benson, J. R.

The Grading of Aggregates for Bituminous Construction—Summary of Proceedings of the Symposium of Mineral Aggregates, 1128.

Berk, A. A.

Discussion, 906, 937.

Berk, Sigmund

Fungus Resistance of Treated Vegetable Fiber Gaskets. Published in ASTM BULLETIN, No. 150, January, 1948, p. 46 (TP18).

Betz, L. D.

Discussion, 906.

Blank, A. I.

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form, 709. Discussion, 736.

Bloem, Delmar L.

Effect of Drying Time on Results of Magnesium Sulfate Soundness Tests. Published in ASTM BULLETIN, No. 152, May, 1948, p. 76 (TP128).

Bobrowsky, A. R.

Discussion, 589, 663.

Bonnet, Frederic

The Five Outstanding Developments in Textiles During the Last Decade—A Resume (condensed Version). Published in ASTM BULLETIN, No. 155, December, 1948, p. 16.

Flammability of Textiles for Garment Purposes—Symposium on Flammability, Published in ASTM BULLETIN, No. 150, January, 1948, p. 29 (TP1).

Bonwitt, W. F.

Quantitative Evaluation of Corrosive Conditions. Published in ASTM BULLETIN, No. 151, March, 1948, p. 84 (TP92); Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 45 (TP203).

Bouvet, Rene

Application of Synthetic Fibers, Including Glass, in the Textile Industry (condensed version) Published in ASTM BULLETIN, No. 150, January, 1948, p. 20.

Bowden, R. C., Jr.

Discussion, 645.

Bowers, Raymon

A Review of Tractor Requirements as Related to Fuels—Summary of Proceedings of Forum on Tractor Fuels, 1215.

Boyd, T. A.

If Doctor Marburg Came Back Today—Annual Address by the President, 26. Published in ASTM BULLETIN, No. 153, August, 1948, p. 55 (TP147).

Research—Everybody's Doing it Now. Published in ASTM BULLETIN, No. 152, May, 1948, p. 53 (TP105).

Briggs, T. H.

European Practice in the Manufacture of Cathodes (condensed version). Published in ASTM BULLETIN, No. 152, May, 1948, p. 39.

Brunstrum, L. C.

Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 97 (TP189).

The Light-Weight Cone for Penetration of Soft Greases. Published in ASTM BULLETIN, No. 154, October, 1948, p. 66 (TP224).

Buechner, W. W.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM BULLETIN, No. 155, December, 1948, p. 54 (TP262).

Burghoff, H. L.

Fatigue Properties of Some Coppers and Copper Alloys in Strip Form, 709.

Burmister, Donald M.

The Importance and Practical Use of Relative Density in Soil Mechanics, 1249.

Burns, Jay R.

Discussion, 754.

Burns, Robert

Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 49 (TP207).

Burrill, E. A.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM BULLETIN, No. 155, December, 1948, p. 54 (TP262).

C

Callendar, L. H.

Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 59 (TP151).

Carpenter, Arthur W.

Discussion, 885.

Carpenter, C. A.

Mineral Aggregates for Bituminous Construction—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Carr, W. J., Jr.

A New D-C Permeameter—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Cavanagh, P. E.

Magnetic Stress Analysis—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Discussion, 763.

Cavanagh, W. R.

Apparatus and Factors in Salt Fog Testing, 153.

Chisholm, Harry E.

New Type Weighted Needles for Determining the Setting Time of Magnesium Oxychloride Cements. Published in ASTM BULLETIN, No. 154, October, 1948, p. 37 (TP195).

Chollar, Robert G.

Metals and Plastics. Published in ASTM BULLETIN, No. 152, May, 1948, p. 80 (TP132).

Clark, Frank, M.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil, 1201. Discussion 1213.

Clarke, Frank E.

Discussion, published in ASTM BULLETIN, No. 150, January, 1948, 49.
Discussion, 926.

Clogg, Mason, Jr.

Color Metallography Simplified—Summary of Proccedings of the Symposium on Metallography in color, 942.

Coburn, M.

Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 77 (TP39).

Cohen, P.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method, 1269. Discussion, 1291.

Collegeman, S. M.

Factors affecting Simulated Service Tests of Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Greases, 1214.

Collins, W. Leighton

Fatigue and Static Load Tests of an Austenitic Cast Iron at Elevated Temperatures, 696.

Connor, C. C.

Factors in the Resistance of Brick Masonry Walls to Moisture Penetration, 1020. Discussion, 1050.
Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 45 (TP203).

Copson, H. R.

Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels, 591. Discussion, 609.
Discussion, 645.

Corey, R.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method, 1269. Discussion, 1291.

Corey, Richard C.

Corrosion of High-Pressure Steam Generators: Status of our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam Generating Tubes—Panel Discussion on Corrosion of Pressure Vessels, 907. Discussion, 937.

Craver, J. Kenneth

The Mechanism of Plasticization in Plastics. Published in ASTM BULLETIN, No. 152, May, 1948, p. 90 (TP142). Discussion, p. 92 (TP144).

Cross, B. J.

Discussion, 932.

Cross, Howard C.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing, 555.

D

Darsey, V. M.

Apparatus and Factors in Salt Fog Testing, 153.

Davis, R. E.

Lightweight Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.
Discussion, 1064.

Decker, J. M.

Discussion, 936.

Dienes, G. J.

An Improved Low-Temperature Brittleness Test. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204).

Dieterly, D. C.

D-C Permeability Testing of Epstein Samples with Double-Lap Joints—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Dietz, A. G. H.

Effect of Speed of Test Upon Strength Properties of Plastics—Symposium on Speed of Testing, 1160. Discussion, 1188.

Ditton, E. N.

Service Wearing Tests. Published in ASTM BULLETIN, No. 152, May, 1948, p. 93 (TP145).

Dodge, H. F.

Introduction—Symposium on Usefulness and Limitations of Samples, 857.
Discussion, 884

Dolan, T. J.

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel, 664. Discussion, 693.
Discussion, 735, 764.

Dollins, C. W.

Discussion, 825.

Donworth, R. B.

Station Design and Material Composition as Factors in Boiler Corrosion—Panel Discussion on Corrosion of Pressure Vessels, 897. Discussion, 905
Discussion, 927.

Dorn, Charles W.

The Retailer's Viewpoint on the Flammability of Textiles—Symposium on Flammability. Published in ASTM BULLETIN, No. 150, January, 1948, p. 36 (TP8).

Droegemueller, E. A.

Better Lead Scavenging Needed for Aviation Gasoline. Published in ASTM BULLETIN, No. 154, October, 1948, p. 53 (TP211).

Duke, James B.

Some Characteristics of Residual Stress Fields During Dynamic Stressing Above The Endurance Limit, 755. Discussion, 765.

E

Ehrke, Louis F.

One-Millionth Second Radiography and Its Development. Published in ASTM BULLETIN, No. 150, January, 1948, p. 59 (TP31)

Eickhoff, Arnold J.

Report on the Evaluation of Surface Treatment of Steel Prior to Paint. Published in ASTM BULLETIN, No. 150, January, 1948, p. 77 (TP49).

Eisen, I.

Quantitative Evaluation of Corrosive Conditions. Published in ASTM BULLETIN No. 151, March, 1948, p. 84 (TP92).

Ellinger, G. A.

Laboratory Corrosion Tests of Iron and Steel Pipes, 618.

Elliott, W. S.

Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 78 (TP38).

Ellis, O. W.

Discussion, 855.

Ellis, W. C.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead, 801. Discussion, 833.

Emch, P. I.

Requirements of an Engine Antifreeze and Methods of Evaluation. Published in ASTM BULLETIN, No. 154, October, 1948, p. 57 (TP215).

Erickson, E. C. O.

A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range. Published in the ASTM BULLETIN, No. 153, August, 1948, p. 84 (TP176).

Everett, H. E.

Tractor Statistics as Related to the Farmer—Summary of Proceedings of the Forum on Tractor Fuels, 1215.

Everitt, W. T.

Service Experience with Grease—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Greases, 1214.

F

Faigen, Harry L.

A Visual Rating System for Rusted Steel Specimens. Published in ASTM BULLETIN, No. 154, October, 1948, p. 39 (TP197).

Feshbach, H.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM BULLETIN, No. 155, December, 1948, p. 54 (TP262).

Findley, W. N.

The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by the Hyperbolic-Sine Law and Activation Energy Theory, 1217.

Finn, W. J.

Laboratory Performance Tests for Antifriction Bearing Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Grease, 1214.

Fishburn, C. C.

Discussion, published in ASTM BULLETIN, No. 156, p. 77 (TP39).

Discussion, 1046.

Fletcher, E. E.

The Application of Color Photography to the Study of NonMetallic Inclusions—Summary of Proceedings of the Symposium on Metallography in Color, 942.

Foote, J. H.

Discussion, 935.

Forgeng, W. D.

Some Applications of Color—Summary of Proceedings of the Symposium on Metallography in Color, 942.

Freeman, J. W.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing, 555. Discussion, 590.

Fried, M. L.

Notch Bar Tension Tests on Annealed Carbon Steel Specimens of Various Sizes and Contours—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Fry, Lawford H.

Testing Speed Limitations for Committee A-1 Specifications for Steel—Symposium on Speed of Testing, 1130.

Fuller, C. S.

Discussion, published in ASTM BULLETIN, No. 152, May, 1948, p. 92 (TP144).

G

Gailus, W. J.

Effect of Speed of Test Upon Strength Properties of Plastics—Symposium on Speed of Testing, 1160. Discussion, 1188.

Gallagher, J. M.

A Method for the Routine Determination of Dry Hiding Power of White Paints. Published in ASTM BULLETIN, No. 153, August, 1948, p. 77 (TP169).

Gause, G. R.

The Amount of Inspection as a Function of Control of Quality—Symposium on Usefulness and Limitations, 886. Discussion, 894.

Gloor, W. E.

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Godfrey, Douglas

Changes Found on Reciprocated Steel, Chromium Plates and Cast Iron Sliding Surfaces, 841.

Gohn, G. R.

The Effect of Small Percentages of Silver and Copper on the Creep Characteristics of Extruded Lead, 801. Discussion, 833.
Discussion, 733.
Discussion, published in ASTM BULLETIN, No. 155, December, 1948, p. 35 (TP243).

Goldbeck, A. T.

Mineral Aggregates for Railroad Ballast—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.
Discussion, 1018.

Goldblum, K. B.

A New Interpretation of Heat Distortion Data of Plastics. Published in ASTM BULLETIN, No. 150, January, 1948, p. 55 (TP27).

Gonnerman, H. F.

Discussion, 971.

Good, J. N.

Changes Found on Reciprocated Steel, Chromium Plate, and Cast Iron Sliding Surfaces, 841.
Discussion, 854.

Goshorn, John C.

An Instrument for Determining the Volume and Bulk Density of Granular Materials. Published in ASTM BULLETIN, No. 151, March, 1948, p. 55 (TP63).

Gothard, N. J.

Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 98 (TP190).

Gregg, L. E.

Discussion, 1017.

Green, D. H.

Requirements of an Engine Antifreeze and Methods of Evaluation. Published in ASTM BULLETIN, No. 154, October, 1948, p. 57 (TP215); Discussion, p. 65, (TP223).

Greenwood, J. Neill

Discussion, 831.

Gross, J.

Measurement of Transparency of Surface Coating. Published in ASTM BULLETIN, No. 153, August, 1948, p. 73 (TP165).

Gross, William E.

An Instrument for Determining the Volume and Bulk Density of Granular Materials. Published in ASTM BULLETIN, No. 151, March, 1948, p. 55, (TP63).

Gunn, W. T.

Petroleum Technology (condensed version). Published in ASTM BULLETIN, No. 151, March, 1948, p. 97 (TP105).

H

Haase, H. F.

Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 45 (TP203).

Hall, A. M.

The Application of Color Photography to the Study of NonMetallic Inclusions—Summary of Proceedings of the Symposium on Metallography in Color, 942.

Halperin, Herman

Discussion, 830.

Hamburger, Walter J.

The Stress-Strain Diagram as a Tool in Textile Research, Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 491.

Hanna, W. C.

Discussion, 959.
Discussion, 971.
Discussion, 1063.
Discussion, 1104.

Hansen, W. C.

Discussion, 1104.

Harder, O. E.

Discussion, 589.

Hardesty, J. M.

Discussion, 1049.

Harvey, C. E.

The Spectrochemical Analysis of Cements and Other Mineral Products, 944.

Hasler, M. F.

The Spectrochemical Analysis of Cements and Other Mineral Products, 944. Discussion, 960.

Herbst, M.

Laboratory Performance Tests for Antifriction Bearing Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.

Hiers, George O.

Discussion, 837.

Hill, Harry

A Versatile Calorimeter for Specific Heat Determinations. Published in ASTM BULLETIN, No. 151, March, 1948, p. 88 (TP96).

Hinkle, C. N.

The Effect of State Tax Regulation and State Specifications on Tractor Fuels from the Petroleum Industry Standpoint—Summary of Proceedings of Forum on Tractor Fuels, 1215.

Holaday, W. M.

The Efficient Production and Utilization of Motor Gasoline—Summary of Meeting on Petroleum Products as Related to Automotive Equipment, 1216.

Hollady, E. L.

Discussion, 883.

Holtz, W. G.

The Determination of Limits for the Control of Placement Moisture in High Rolled-Earth Dams, 1240.

Hoppmann, William H., II

Effect of Fatigue on Tension-Impact Resistance. Published in ASTM BULLETIN, No. 155, December, 1948, p. 36 (TP244); Discussion, 38 (TP246).

Howell, F. M.

A Method of Calibrating Extensometers. Published in ASTM BULLETIN, No. 155, December, 1948, p. 33 (TP241), Discussion, 35 (TP243).
Discussion, 1200.

Hunter, Richard S.

The Gloss Measurement of Paint Finishes. Published in ASTM BULLETIN, No. 150, January, 1948, p. 72 (TP44).

Hyde, L. K.

Methods and Equipment for Controlling Speed of Testing—Symposium on Speed of Testing, 1191.

I

Imbembo, E. A.

Notched-Sensitivity of Ship-Plate—Correlation of Laboratory-Scale Tests with Large-Scale Plate Tests—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Insley, Herbert

Discussion, 1126.

J

Jackson, F. H.

Discussion, 970.

Jacobsen, A. E.

Mechanical Operation of the Bird Film Applicator. Published in ASTM BULLETIN, No. 151, March, 1948, p. 95 (TP103).

Jain, B.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method, 1269. Discussion, 1291.

Jensen, H. S.

Mechanical Operation of the Bird Film Applicator. Published in ASTM BULLETIN, No. 151, March, 1948, p. 95 (TP103).

Johanson, Stig

The Preparation of Uniform Films and Their Dependence on the Irregularities of the Base Surface. Published in ASTM BULLETIN, No. 155, December, 1948, p. 53 (TP261).

Jominy, W. E.

Discussion, 692.

Jonakin, J.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method, 1269. Discussion, 1291.

Jones, P. G.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals, 648. Discussion, 663.

K

Kahn, Frank

Influence of Small Percentages of Silver on the

Tensile Strength of Extruded Lead Sheathing, 815. Discussion, 837.

Kahn, N. A.

Notched-Sensitivity of Ship-Plate—Correlation of Laboratory-Scale Tests with Large-Scale Plate Tests—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Kanter, J. J.

Stability of Steels as Affected by Temperature (Project No. 29). Appendix, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Katz, S. H.

Nontoxic Rubber for Gas Marks, 1293.
Discussion, 1299.
Discussion, 885.

Kaufman, Gus.

Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 97 (TP189).
Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 65 (TP223).

Kelley, J. A., Jr.

Mineral Aggregates for Low-Cost Roads and Water-Bound Macadams—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Kelly, J. W.

Lightweight Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Kienle, R. H.

Measurement of Transparency of Surface Coatings, published in ASTM BULLETIN, No. 153, August, 1948, p. 73 (TP165).

Kiszka, John C.

What's in an Alloy? Published in ASTM BULLETIN, No. 151, March, 1948, p. 51 (TP59).

Knapp, J. C.

Discussion, 882.

Knowlton, H. B.

Discussion, 690.

Korb, E. L.

Induction Period Calculator. Published in ASTM BULLETIN, No. 153, August, 1948, p. 99 (TP191).

Kosting, P. R.

Discussion, 692.

Kratzer, J. C.

Requirements of an Engine Antifreeze and Methods of Evaluation. Published in ASTM BULLETIN, No. 154, October, 1948, p. 57 (TP215).

Kriege, H. F.

Mineral Aggregates in the Chemical and Processing Industries and in Certain Other Uses—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

L

Lace, Robert H.

Discussion, 876.
Discussion, 894.

Lamb. J. J.

Tensile Stress-Strain Relationships of Laminated Plastics for Small Strains. Published in ASTM BULLETIN, No. 151, March, 1948, p. 59 (TP67).

Lamson, Horatio W.

The Evaluation of Hysteresis Core Loss by Power Equations—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Langer, B. F.

Discussion, 799.

Lankford, W. T.

Hydraulic Bulge Testing of Sheet Metals—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

La Que, F. L.

Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 44 (TP202).
Discussion, 608

Larson C. M.

Petroleum Lubricating Oils as Related to Automotive Equipment—Summary of Meeting on Petroleum Products as Related to Automotive Equipment, 1216.

Lessells, John M.

Discussion, 690.
Discussion, 765.
Discussion, 799.
Discussion, 854.

Leutriz, John, Jr.

The Effect of Fungi and Humidity on Plastics. Published in ASTM BULLETIN, No. 152, May 1948, p. 88 (TP140).

Liander, Halvard

Discussion, published in ASTM BULLETIN, No. 154, October, 1948, p. 45 (TP203).

Liska, J. A.

Speed of Testing of Wood: Factors in its Control and its Effect on Strength—Symposium on Speed of Testing, 1139.

Little, R. W.

Flameproofing in Theory and Practice—Symposium on Flammability, Published in ASTM BULLETIN, No. 150, January, 1948, p. 34 (TP6).

Loveland, R. P.

Quality and Quantity of Illumination in Color Metallography—Summary of Proceedings of the Symposium on Metallography in Color, 942.

Low, J. R., Jr.

Measurement of Ductility in Sheet Metals—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Lutz, W. H.

Paint—Servant of Industry (condensed vervion). Published in ASTM BULLETIN, No. 150, January, 1948, p. 14.

Lykken, Louis

Photoelectric Measurement of a Color Index of Liquid Petroleum Products. Published in ASTM BULLETIN, No. 154, October, 1948, p. 68 (TP226).

M

Magee, William P.

Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing, 815.
Discussion, 837.

Majors, Harry, Jr.

Change in Hardness of a Metal Bar Under Low Cycles of Reversed and Pulsating Plastic Bending. Published in ASTM BULLETIN, No. 155, December, 1948, p. 39 (TP247).

Mantz, V. M.

Discussion, published in ASTM BULLETIN, No. 150, January, 1948, p. 49 (TP21).

Markwardt, L. J.

Speed of Testing of Wood: Factors in its Control and Its Effect on Strength—Symposium on Speed of Testing, 1139.

Marzolf, S. B.

Laboratory Corrosion Tests of Iron and Steel Pipes, 618.

Mather, Bryant

Petrographic Indentification of Reactive Constituents in Concrete Aggregate—Symposium on Reactive Materials in Concrete, 1120.
Discussion, 1126.
Discussion, 1017.
Discussion, 1104.

Maynard, C. E.

Permanent Magnet Testing Methods and Their Validity in Determining Product Performance—Summary of Proceedings of the Symposium on Magnetic Testing, 943

MacKenzie, J. T.

Discussion, 855.

McBurney, J. W.

Discussion, 1047.

McCormack, Robert L.

A Standard Diode for Radio Tube Cathode-

Core-Material Approval Tests (condensed version). Published in ASTM Bulletin, No. 152, May, 1948, p. 37.

McCoy, W. J.

Discussion, 959.

McDonald, John C.

Tensile, Creep and Fatigue Properties at Elevated Temperatures of Some Magnesium-Base Alloys, 737.

McIntosh, L. R.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM Bulletin, No. 155, December, 1948, p. 54 (TP262).

McKee, S. A.

A Worker-Consistometer for Lubricating Greases. Published in ASTM Bulletin, No. 153, August, 1948, p. 90 (TP182); Discussion, p. 98 (TP190).

McKinney, D. D.

Discussion, 906.

McLaren, A. D.

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers. Published in ASTM Bulletin, No. 155, December, 1948, p. 50 (TP258).

McLennan, L. W.

The Thixotropic Characteristics of Lubricating Oil Greases. Published in ASTM Bulletin, No. 152, May, 1948, p. 71 (TP123).

McMaster, Robert C.

An Investigation of the Possibilities of Organic Coatings for the Prevention of Premature Corrosion-Fatigue Failures in Steel, 628. Discussion, 645.

Meara, F. L.

Discussion, published in ASTM Bulletin, 153, August, 1948, p. 81 (TP173).

Meissner, Harmon S.

Discussion, 1064.
Discussion, 1104.
Discussion, 1115.

Meissner, H. P.

Some Factors Affecting Adhesive Bond Formation. Published in ASTM Bulletin, No. 151, March, 1948, p. 80 (TP88).

Melsheimer, L. A.

Measurement of Transparency of Surface Coatings. Published in ASTM Bulletin, No. 163, August, 1948, p. 73 (TP165).

Merrill, E. W.

Some Factors affecting Adhesive Bond Formation. Published in ASTM Bulletin, No. 151, March, 1948, p. 80 (TP88).

Mielenz, R. C.

Petrographic and Mineralogic Characteristics of Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.
Tests Used by the Bureau of Reclamation for Indentifying Reactive Concrete Aggregated—Symposium on Reactive Materials in Concrete, 1071.
Discussion, 1105.

Miller, R. F.

Measurement of Volume Resistivity of Electrically Conducting Rubber-Like Material. Published in ASTM Bulletin, No. 151, March, 1948, p. 91 (TP99).

Misenhelder, P. D.

Discussion, 1017.

Mitch, J. E.

Permanent Magnet Testing Methods and Their Validity in Determining Product Performance—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Moody, L. S.

A New Intrepretation of Heat Distortion Data of Plastics. Published in ASTM Bulletin, No. 150, January, 1948, p. 55 (TP27).

Moore, A. A.

The Effect of Speed of Testing on Magnesium-Base Alloys—Symposium on Speed of Testing, 1133.

Tension Testing of Magnesium Alloy Tubing, 205.
Discussion, published in ASTM BULLETIN, No. 155, December, 1948, p. 35 (TP243).

Moreton, D. H.

Development of Functional Grease Test Methods for the Aircraft Industry—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.

Moser, Frank

Some Fundamentals of Glass Adhesion. Published in ASTM BULLETIN, No. 150, January, 1948, p. 51 (TP23).

Moulton, H. R.

Plastics as Optical Materials. Published in ASTM BULLETIN, No. 151, March, 1948, p. 75 (TP83).

Muhlenbruch, C. W.

The Effect of Repeated Loading on the Bond Strength of Concrete—II, 977.
Discussion, 986.
Tests of 77-Year-Old Wrought Iron (condensed version). Published in ASTM BULLETIN, No. 151, March, 1948, p. 44.

N

Nason, H. K.

Discussion, 1187.

Newkirk, Burt L.

Discussion, 763.

Newmark, N. M.

An Hypothesis for the Determination of Cumulative Damage in Fatigue, 767. Discussion, 800.

Noble, T. Walter

Low-Pressure Moldings and Laminates. Published in ASTM BULLETIN, No. 151, March, 1948, p. 77 (TP85).

O

Oldacre, William H.

Water-Mixed Cutting Fluids—Symposium on Cutting Oils. Published in ASTM BULLETIN, No. 151, March, 1948, p. 45 (TP53).

Orning, A. A.

Discussion, 1290.

Owens, F. R.

Discussion, 936.

P

Parsons, D. E.

Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 76 (TP38).

Parker, W. E.

Discussion, 972.

Pauls, J. T.

Mineral Aggregates for Bituminous Construction—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Perry, A. N.

The Use of a Test Track in Developing Good Packaging and Carloading Methods. Published in ASTM BULLETIN, No. 155, December, 1948, p. 38 (TP246).

Peterson, R. E.

Discussion, 690.

Phelps, Howard S.

Influence of Small Percentages of Silver on the Tensile Strength of Extruded Lead Sheathing, 815. Discussion, 837.

Piech, F. E.

The Use of the Rossi-Peakes Flow Tester in Measuring the Apparent Viscosity of Plastics at Temperatures from 115 to 175 C. Published in ASTM BULLETIN, No. 151, March, 1948, p. 70 (TP78).

Pilling, N. B.

Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data, 610.

Plummer, H. C.

Discussion, 1049.

Prater, T. A.

Measurement of Ductility in Sheet Metals—

Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Pope, C. L.

Service Experience with Grease—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214.

Service Experience with Turbine Oils—Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, May, 1948, p. 62 (TP114).

Power, George E.

Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 64 (TP156).

Prendergast, W. A.

Laboratory Performance Tests for Antifriction Bearing Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Greases, 1214

Price, W. H.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete, 962.

Discussion, 970.

Grading of Mineral Aggregates for Portland-Cement Concrete and Mortars—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Proudley, C. E.

Sampling of Mineral Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Discussion, 968.

Puryear, O. P.

Laboratory Performance Tests for Antifriction Bearing Greases—Summary of Proceedings of the Symposium on Functional Tests for Ball-Bearing Grease, 1214.

Q

Quackenbos, H. M., Jr.

Discussion, published in ASTM BULLETIN, No. 152, May, 1948, p. 87 (TP139).

R

Raab, Edward L.

The Detection of Corrosive Sulfur Compounds in Mineral Transformer Oil, 1201.

Reehl, E. A.

Cutting Fluids—Symposium on Cutting Oils. Published in ASTM BULLETIN, No. 151, March, 1948, p. 47 (TP55).

Reynolds, A. H.

Discussion, 931.

Reynolds, E. E.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing, 555.

Rhoades, Roger

Petrographic and Mineralogic Characteristics of Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Richart, F. E., Jr.

An Hypothesis for the Determination of Cumulative Damage in Fatigue, 767.

Discussion, 800.

Discussion, 986.

Riegel, G. C.

Discussion, 692.

Robinson, Ernest L.

High Temperature Bolting Materials (Project No. 16), 214. Discussion, 215.

Robison, J. W.

Effect of Delayed Mixing of Prebatched Moist Aggregates and Cement on the Strength and Durability of Concrete, 962. Discussion, 973.

Rockwood, N. C.

Production and Manufacture of Fine and Coarse Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Romer, J. B.

Discussion, 934.

Roop, W. P.

Tests of Ductility in Ship Structure—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Ryan, Joseph D.

Automotive Glazing with Plastics. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

S

Sachs, G.

Notch Bar Tension Tests on Annealed Carbon Steel Specimens of Various Sizes and Contours—Summary of Proceedings of the Symposium on Deformation of Metals as Related to Forming and Service, 896.

Scafe, E. T.

Discussion, 1212.

Schapiro, Leo

Discussion, 589.

Schiefer, Herbert F.

Measurement and Elimination of Inertia Effects in Textile Testing Equipment by Means of Electric Strain Gages. Published in ASTM BULLETIN, No. 150, January, 1948, p. 43 (TP15).

Schmidt, P. L.

Variation of Core Loss and Permeability of Electrical Grade Silicon Sheet Steel—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Schmutz, F. C.

A Method for the Routine Determination of Dry Hiding Power of White Paints. Published in ASTM BULLETIN, No. 153, August, 1948, p. 77 (TP169).

Schofield, L. B.

Discussion, 1212.

Schwarz, Edward R.

The Importance of the Time Factor in the Study of Textile Properties. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 485.

Scott, R. K.

Discussion, 930.

Seiler, Charles J.

Theories of Adhesion as Applied to the Adhesive Properties of High Polymers. Published in ASTM BULLETIN, No. 155, December, 1948, p. 50 (TP258).

Setchkin, N. P.

Discussion, published in ASTM BULLETIN, No. 151, March, 1948, p. 66 (TP74).

Shaw, F. B.

Discussion, 884.

Shinn, D. A.

Discussion, 1190.

Siess, C. P.

Discussion, 986.

Simmons, Ward F.

High-Temperature Properties of Rotor Disks for Gas Turbines as Affected by Variables in Processing, 555.

Simon, Leslie E.

On Variation in Materials, Testing, and Sample Sizes—Symposium on Usefulness and Limitations of Samples, 877.
Discussion, 882.

Slack, Charles M.

One-Millionth Second Radiography and Its Development. Published in ASTM BULLETIN, No. 150, January, 1948, p. 59 (TP31).

Smith, A. A., Jr.

Discussion, 828.

Smith, B. M.

Testing Magnetic Materials—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Smith, E. F.

An Improved Low-Temperature Brittleness Test. Published in ASTM BULLETIN, No. 154, October, 1948, p. 46 (TP204).

Smith, G. H.

The Thixotropic Characteristics of Lubricating Oil Greases. Published in ASTM

Bulletin, No. 152, May, 1948, p. 71 (TP123).

Smith, R. B.

A.S.T.M. Code Systems for Aluminum Alloys and Magnesium Alloys. Published in ASTM Bulletin, No. 151, March, 1948, p. 50 (TP58).

Smith, Theodore R.

A Rapid Method of Testing Materials for The Alkali-Aggregate Reaction—Symposium on Reactive Materials in Concrete, 1108.

Snyder, C. J.

Discussion, 828.

Snyder, E. A.

Discussion, 1212.

Sookne, Arnold M.

Some Applications of Stress-Strain Measurements to the Determination of Fiber Properties. Published in A.S.T.M. Standards on Textile Materials, October, 1948, p. 498.

Sperduto, A.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM Bulletin, No. 155, December, 1948, p. 54 (TP262).

Sprague, J. C.

Discussion, 1105.

Sprague, O. V.

Service Experience with Turbine Oils—Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM Bulletin, No. 152, May, 1948, p. 62 (TP114).

Stamm, Alfred J.

A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range. Published in ASTM Bulletin, No. 153, August, 1948, p. 84 (TP176).

Stanton, Thomas E.

Correlation of Laboratory Tests with Field Experiences of Excessive Concrete Expansion Induced by a Reaction Between the Cement and Aggregate—Symposium on Reactive Materials in Concrete, 1057. Discussion, 1065.

Stearns, E. I.

Measurement of Transparency of Surface Coatings. Published in ASTM Bulletin, No. 153, August, 1948, p. 73 (TP165).

Stern, R. L.

Discussion, published in ASTM Bulletin, No. 153, August, 1948, p. 64 (TP156).

Sticha, E. A.

Stability of Steels as Affected by Temperature (Project No. 29). Appendix, Report of Joint Committee on Effect of Temperature on the Properties of Metals, 239.

Swaine, D. J.

The Action of "Chromic Acid" on Zinc Coatings. Published in ASTM Bulletin, No. 154, October, 1948, p. 52 (TP210).

Discussion, Published in ASTM Bulletin, No. 156, January, 1949, p. 49 (TP11).

Swayze, M. A.

Discussion, 970.

Sweet, Harold S.

Physical and Chemical Tests of Mineral Aggregates and Their Significance—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Research on Concrete Durability as Affected by Coarse Aggregate, 988. Discussion 1018.

Discussion, 1126.

T

Tanenhaus, S. J.

Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter. Published in ASTM Bulletin, No. 154, October, 1948, p. 74 (TP232).

Tatnall, F. G.

Discussion, published in ASTM Bulletin No. 155, December, 1948, p. 35 (TP243).

Taylor, Thomas G.

Determination of the Air Content of Mortars by the Pressure Method. Published in

ASTM BULLETIN, No. 155, December 1948, p. 44 (TP252).

Thilo, E. R.

Frankford Arsenal Experience with High-Speed Radiography. Published in ASTM BULLETIN, No. 150, January, 1948, p. 69 (TP41).

Thurston, R. C. A.

Dynamic Calibration Method Uses Modified Proving Ring. Published in ASTM BULLETIN, No. 154, October, 1948, p. 50 (TP208).

Toulouse, J. H.

Load Concentration in Corrugated Paper. Published in ASTM BULLETIN, No. 152, May, 1948, p. 77 (TP129).

Tremper, Bailey

Correlation of Laboratory Tests with Field Experience in Alkali-Aggregate Reaction—Symposium on Reactive Materials in Concrete, 1067.
Discussion, 1064.

Tuthill, Marshall W.

A Review of the Tin Situation, 146.

V

Van de Graaff, R. J.

An Investigation of Radiography in the Range from 0.5 to 2.5 Million Volts. Published in ASTM BULLETIN, No. 155, December, 1948, p. 54 (TP262).

Voss, Walter C.

Discussion, published in ASTM BULLETIN, No. 156, January, 1949, p. 77 (TP39).
Discussion, 1040.

von Fuchs, G. H.

Discussion, 1211.

W

Waldron, L. J.

Laboratory Corrosion Tests of Iron and Steel Pipes, 618.

Walker, Stanton

Discussion, 1065, 1115.

Walsh, Ernest F.

The Care of the Lubricant and Maintenance of the Lubricating System for Central Station Turbine Equipment—Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, May, 1948, p. 64 (TP116).

Wangelin, D. J.

Correlation of Laboratory Testing of Cutting Fluids With Field Tests—Symposium on Cutting Oils. Published in ASTM BULLETIN, No. 151, March, 1948, p. 48 (TP56).

Warner, A. J.

Electrical Properties of Plastics. Published in ASTM BULLETIN, No. 153, August, 1948, p. 60 (TP152).
Discussion, published in ASTM BULLETIN, No. 153, August, 1948, p. 64 (TP156).

Watkins, George B.

Automotive Glazing with Plastics. Published in ASTM BULLETIN, No. 153, August, 1948, p. 65 (TP157).

Weatherwax, Richard C.

A Means of Determining the Hardness of Wood and Modified Woods Over a Broad Specific Gravity Range. Published in the ASTM BULLETIN, No. 153, August, 1948, p. 84 (TP176).

Wesley, W. A.

Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data, 610.

Whirl, S. F.

Discussion, 905, 926.

White, H. S.

A Worker-Consistometer for Lubricating Greases. Published in ASTM BULLETIN, No. 153, August, 1948, p. 90 (TP182).
Discussion, p. 98 (TP190).

Wiegand, D. E.

The Testing of Magnetic Recording Media—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Wilcock, D. F.

Grease—An Oil Storehouse for Bearings—Summary of Proceedings of the Symposium on Functional Tests for Ball Bearing Grease, 1214.

Wilks, S. S.

Sampling and Its Uncertainties—Symposium on Usefulness and Limitations of Samples, 859. Discussion, 876.

Willis, E. A.

Mineral Aggregates for Low-Cost Roads and Water-Bound Macadams—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Wilson, C. D.

Steam Turbines and Their Lubrication—Symposium on Service Experience with Inhibited Turbine Oils. Published in ASTM BULLETIN, No. 152, March, 1948, p. 66 (TP118).

Winston, Gerald

Report of the A.S.T.M. Task Group Studying the Wyzenbeek Precision Wear Test Meter. Published in ASTM BULLETIN, No. 154, October, 1948, p. 74 (TP232).

Witte, Leslie P.

Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates—Symposium on Reactive Materials in Concrete, 1071. Discussion, 1105.

Wlodek, T.

Magnetic Stress Anlysis—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

Woods, K. B.

Distribution of Mineral Aggregates—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Woolf, D. O.

A Rapid Method of Testing Materials for The Alkali-Aggregate Reaction—Symposium on Reactive Materials in Concrete, 1108. Discussion, 1115.

Discussion, 1104, 1126.

Needed Research—Summary of Proceedings of the Symposium on Mineral Aggregates, 1128.

Worley, W. J.

An Experimental Study of the Influence of Various Factors on the Mode of Fracture of Metals, 648.

The Effect of Temperature on the Creep of Two Laminated Plastics as Interpreted by The Hyperbolic-Sine Law and Activation Energy Theory, 1217.

Y

Yen, C. S.

Some Aspects of the Effect of Metallurgical Structure on Fatigue Strength and Notch-Sensitivity of Steel, 664. Discussion, 693.

Yurenka, S.

Effect of Speed of Test Upon Strength Properties of Plastics—Symposium on Speed of Testing, 1160. Discussion, 1188.

Z

Zuschlag, Theodore

Magnetic Analysis Inspection in the Steel Industry—Summary of Proceedings of the Symposium on Magnetic Testing, 943.

LIST OF SPECIAL TECHNICAL PUBLICATIONS

Since the publication of "The Life and Life Work of Charles B. Dudley" in 1911, the first special publication of the Society, a memorial volume to the first president of A.S.T.M. (1900–1909), the Society has issued many technical books, pamphlets, and charts which have not been included in the *Proceedings*, ASTM BULLETIN, or *Book of A.S.T.M. Standards*. In view of the value of these publications to research workers, engineers, industrialists, and students in specialized fields, on the recommendation of the Administrative Committee on Papers and Publications, these publications have been collected chronologically into an A.S.T.M. Technical Series and given a Special Technical Publication Number. This number can be used in ordering copies.

The Technical Series, a list of which follows, includes all the separate publications issued by A.S.T.M. including those issued jointly with other societies. The Series does not include special publications that are reprints from the *Book of Standards* or *Proceedings*, such as compilations of standards and the several symposiums published in *Proceedings*. The publications in the Technical Series are made available to members at special member prices.

If any members have copies of publications indicated as being out of stock and would be willing to sell them back to the Society we should indeed be pleased to be so advised.

Special Technical Publication Number	Title
†1	The Life and Life Work of Charles B. Dudley (Memorial Volume) (1911).
†2	Selected Standard Specifications for Steel and Steel Products (In English, German, French, and Spanish) (1912).
†3	Reports of the Committee on Preservative Coatings for Structural Materials (1914).
†4	Data Considered by Committee C-1 in Preparing the Standard Specifications and Tests for Portland Cement (First and Second Reports of the Joint Conference on Uniform Methods of Tests and Standard Specifications for Cement) (1919).
†5	Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete (1924).
5-A	Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete, Recommended Practice and Standard Specifications for Concrete and Reinforced Concrete (Published with and without Supplement) (1940).
†6	Annotated Bibliography on Impact Testing (Special Mimeograph pamphlet).
7	The Significance of Tests of Petroleum Products (Reprinted from 1928 Proceedings but included here because it represents the first of a series).
7-A	The Significance of Tests of Petroleum Products (1934).
†8	Glossary of Definitions of Terms Appearing in A.S.T.M. Standards and Tentative Standards (Special Mimeograph pamphlet) (1929).
†9	Abstracts of Articles on Fatigue of Metals Under Repeated Stress Appearing in the Technical Press from July 1, 1928 to June 30, 1929 (1929).
†9-A	Abstracts of Articles on Fatigue of Metals Under Repeated Stress Appearing in the Technical Press from July 1, 1929 to June 30, 1930 (1931).
†10	Symposium on Developments in Automotive Materials (Detroit Regional Meeting, March 19, 1930).
†11	Symposium on Welding (Pittsburgh Regional Meeting, March 18, 1931).
†12	Symposium on Effect of Temperature on Metals (1931).
†13	Symposium on Rubber (Cleveland Regional Meeting, March 2, 1932).
†14	Joint Committee on Test Methods and Forms (1933).
15	A.S.T.M. Manual on Presentation of Data (With Tables of Squares and Square Roots) (1933).
15-A	Second printing — Including Supplements A and B (1937).

---

† Out of stock.

Special Technical Publication Number	Title
15-B	Third printing (1940).
†16	Symposium on Motor Lubricants (New York Regional Meeting, March 8, 1933).
†17	Symposium on the Outdoor Weathering of Metals and Metallic Coatings (Washington Regional Meeting, March 7, 1934).
†18	Symposium on Paint and Paint Materials (Philadelphia Regional Meeting, March 6, 1935).
†19	Discussion on the Significance of "Yield Strength" of Metals in Design and Specifications (Mimeographed) (1935).
20	Symposium on the Place of Materials in Automobile Roads and Rides (1935).
†21	The Role of Materials in Modern Housing (1935).
22	Report on Significance of Tests of Concrete and Concrete Aggregates (1935).
†22-A	Report on Significance of Tests of Concrete and Concrete Aggregates (Second Edition) (1943).
†23	Symposium on Industrial Fuels (1936).
24	Symposium on High-Strength Constructional Metals (Pittsburgh Regional Meeting, March 4, 1936).
†25	Symposium on Pearlitic Malleable Cast Iron (1936).
†26	Data on Nominal Creep Strength of Steels at Elevated Temperatures (1936).
27	Proposed Methods of Analysis of Industrial Waters (1936).
†28	Symposium on Radiography and X-ray Diffraction (1936).
28-A	Symposium on Radiography (1943).
29	Symposium on Lubricants (Chicago Regional Meeting, March 3, 1937).
†30	Symposium on Wear of Metals (1937).
31	Viscosity-Temperature Charts (1937).
†32	Symposium on Corrosion Testing Procedures (1937).
33	Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings (1937).
†34	Symposium on Consistency (1937).
†35	Symposium on Plastics (Rochester Regional Meeting, March 9, 1938).
†36	Symposium on Protecting Metals Against Corrosion (1938).
37	Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys (1938).
†38	Standard Specifications for Gypsum Plastering (Published Jointly with the American Institute of Architects) (1938).
39	Symposium on Thermal Insulating Materials (Columbus Regional Meeting, March 8, 1939).
40	Symposium on Lime (Columbus Regional Meeting, March 8, 1939).
41	Index to the Literature on Spectrochemical Analysis 1920–1937 (1939).
41-A	Index to the Literature on Spectrochemical Analysis 1920–1939 (1941).
41-B	Index to the Literature on Spectrochemical Analysis, Part II, 1940–1945 (1947).
42	Viscosity Index Tables (1940).
43	Conversion Tables for Kinematic and Saybolt Universal Viscosities (1940).
44	Symposium on New Materials in Transportation (Detroit Spring Meeting, March 6, 1940).
45	Evaluation of Petroleum Products (1940).
†46	Topical Discussion on Radiographic Testing of Airplane Components (1941).
†47	Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures. Prepared by H. W. Gillett (1941).
48	X-ray Diffraction Data Cards for Chemical Analysis (1941) Index, 1941. Second Index, 1945.
48-A	Supplementary X-ray Diffraction Cards (1944). Index, 1945.
49	Cotton Yarn Appearance Standards (1941).
50	Symposium on Color (Washington Spring Meeting, March 5, 1941).
51	Symposium on New Methods for Particle Size Determination in the Subsieve Range (Washington Spring Meeting, March 4, 1941).
52	Tables of Data on Chemical Composition, Physical and Mechanical Properties of Wrought Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Steels (1942).
53	Hardenability of Steel (Chart) (1943).
53-A	Hardenability of Steel (Chart) (1946).

SPECIAL TECHNICAL PUBLICATION NUMBER	TITLE
54	Symposium on Paint (Buffalo Spring Meeting, March 3, 1943).
55	Symposium on Powder Metallurgy (Buffalo Spring Meeting, March 3, 1943).
56	Bibliography and Abstracts on Electrical Contacts (1943).
56-A	1944 Supplement to the Bibliography and Abstracts on Electrical Contacts.
56-B	1945 Supplement to the Bibliography and Abstracts on Electrical Contacts.
56-C	1946 Supplement to the Bibliography and Abstracts on Electrical Contacts (1946).
56-D	1947 Supplement to the Bibliography and Abstracts on Electrical Contacts (1947).
†57	Symposium on Mildew Resistance (1943).
58	Report on Standard Samples for Spectrographic Analysis by W. R. Brode and B. F. Scribner (1944).
58-A	Report on Standard Samples for Spectrochemical Analysis, Compiled by E-2, Sub. IV (1947).
59	Symposium on Plastics (1944).
60	Paper and Paperboard, Characteristics, Nomenclature, and Significance of Tests (1944).
61	Symposium on the Applications of Synthetic Rubbers (1944).
62	Symposium on Magnetic Particle Testing (1945).
63	Report on Behavior of Ferritic Steels at Low Temperatures by H. W. Gillett and Francis T. McGuire (Part I text, Part II Illustrations) (1945).
64	Symposium on Stress-Corrosion Cracking of Metals (Published Jointly with the American Institute of Mining and Metallurgical Engineers) (1945).
65	Symposium on Adhesives (1946).
66	Discussion on Statistical Quality Control in Its Application to Specification Requirements (Pittsburgh Spring Meeting, February 26, 1946).
67	Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals (Pittsburgh Spring Meeting, February 26, 1946).
68	Symposium on Materials for Gas Turbines (1946).
69	Symposium on Oil Procurement Practices (1946).
70	Symposium on Bearings (1947).
71	Forum on Diesel Fuel Oils (1947).
72	Symposium on Testing of Parts and Assemblies (1947).
73	Symposium on pH Measurement (1947).
74	Symposium on Rubber Testing (1947).
75	Symposium on Paint and Paint Materials (1947).
76	Symposium on Spectroscopic Light Sources (1947).
77	Symposium on Synthetic Lubricants (1947).
78	Symposium on Effects of Low Temperature on Materials (1947).
79	Symposium on Load Tests of Bearing Capacity of Soils (1947).
80	Number assigned but not used.
81	Petroleum Products as Related to Automotive Equipment (1947).
82	Forum on Tractor Fuels (1948).
83	Symposium on Mineral Aggregates (1948).
84	Symposium on Functional Tests For Ball-Bearing Greases (1948).
85	Symposium on Magnetic Testing (1948).
86	Symposium on Metallography in Color (1948).
87	Symposium on Deformation of Metals as related to Forming and Service (1948).